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DEVELOPMENT AND EVALUATION OF POLYIMIDE LAMINATES FOR PRINTED WIRING BOARD APPLICATIONS

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16. ABSTRACT

This report describes a developmental and evaluation program on polyimide laminates as printed wiring boards. The development of an experimental polyimide with low linear coefficient of thermal expansion normal to the plane of the board is discussed, and its properties are evaluated along with two commercial polyimide printed wiring boards for comparison with the type G-10 epoxy board. Thermal cycling data indicate an urgent need for effectively controlling the expansion normal to the plane of the board in order to minimize solder joint damage.

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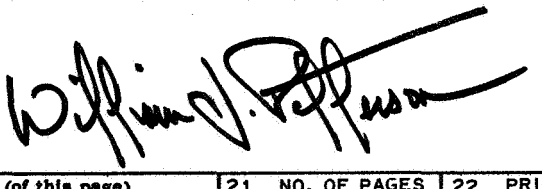

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DEVELOPMENT AND EVALUATION OF POLYIMIDE LAMINATES FOR PRINTED WIRING BOARD APPLICATIONS

SUMMARY

The efforts of this program have been directed toward development of a controlled expansion printed wiring board based on a polyimide laminate. The basic construction of the board consists of three layers of tridirectionally woven cloth impregnated with alumina-filled polyimide resin. Faced on each side of this is a single layer of scrim cloth followed by copper cladding.

The experimental board and two commercial polyimides have been evaluated in terms of processing as well as electrical, physical, and mechanical properties for comparison with type G-10 epoxy. Thermal cycling tests have been carried out from -55 to +100°C for 500 cycles to determine incidence of cracking as a function of board type and joint configuration. The overall solder joint cracking caused by the experimental board was significantly lower than that for either the epoxy or the commercial polyimide boards.

INTRODUCTION

In recent years the designers and users of high reliability electronic circuits have been faced with the recurring problem of failures in printed wiring boards (PWB). Many of the electronic hardware problems in the Saturn program could be related to solder joint cracking and a host of modifications were incorporated, including stress relief bends and plated-through holes, to overcome the cracked joints. The origin of cracking is related directly to the inherent expansion characteristics of the materials comprising the solder joint. The difference in linear coefficient of thermal expansion between the metallic and nonmetallic elements affecting the solder joint is quite significant. The stresses on the PWB joints during thermal cycling arise primarily from two sources: (1) The conformal coating applied over the PWB surface which has a high linear coefficient of thermal expansion and transmits stress through the component lead to the joint, and (2) The relatively high coefficient of thermal expansion of the board material itself in the axis normal to the plane of the board. This report concerns the latter problem and describes the development of a new polyimide board to overcome this deficiency, together with evaluation of several candidate polyimide PWB materials.

BASIC MATERIAL CONSIDERATIONS

The most commonly used PWB substrate in the electronics industry is the epoxy-fiberglass laminate. The direction of orientation of the glass reinforcement is in the plane of the board giving very good control of expansion coefficient (α) in that axis. However, there is no reinforcement normal to the plane of the board and this is manifested in a much higher α-value in the thickness direction as illustrated in Figure 1. Here it is seen that α in the plane of the board is approximately 15 x 10^{-6} in/in-°C at 50°C, as opposed to 60 x 10^{-6} normal to the plane of the board. Thus, the expansion anisotropy in the board is at least four-fold. This is a critical value, since electronic component leads extend through the board to be terminated on the other side by a solder joint and the lead orientation is normal to the plane of the board. The stresses generated by the high α-value of the board can be transmitted directly to the restraining solder joint. Figure 1 also emphasizes the presence of a glass transition (Tg) at 95 - 100°C above which the α-value increases exponentially. This phenomenon is characteristic of materials such as epoxies and further aggravates the expansion problem in the region above 75°C.

The geometry of a typical PWB joint assembly is shown in Figure 2. In this case there is the additional feature of the plated-through hole. In preparation of this joint the lead and general hole area are heated by the iron in order to flow the solder on the lead termination and through the plated-through hole. The adjacent board material is heating up accordingly, but with its significantly higher α-value normal to the board plane, it is expanding at a faster rate than the metallic components of the joint. The solder has now flowed through the hole and the iron is removed to allow the joint to solidify. The board is still in an expanded condition when the joint solidifies so that some residual stress is inescapably developed. Subsequent thermal cycling continues to stress the joint until cracking occurs, as shown in Figure 3, looking down on the top of the solder joint. Here, a crack has propagated around the lead and can result in electrical isolation of the component lead from the conductor pattern.

Table 1 compares the α-values for various PWB joint materials, showing the large disparity between the epoxy board and the other constituents. At this point, the polyimide-based laminate appeared to offer a potential solution to the problem. The expansion characteristics of a commercial polyimide/fiberglass laminate are also given in Table 1. The polyimide board has an inherently lower α-value normal to the plane of the board than the epoxy. Also, the mechanical, thermal and electrical properties of the polyimide material are generally quite good. It has the additional advantage of no glass transition (Tg) in the expected service range of the printed wiring boards. Thus, this

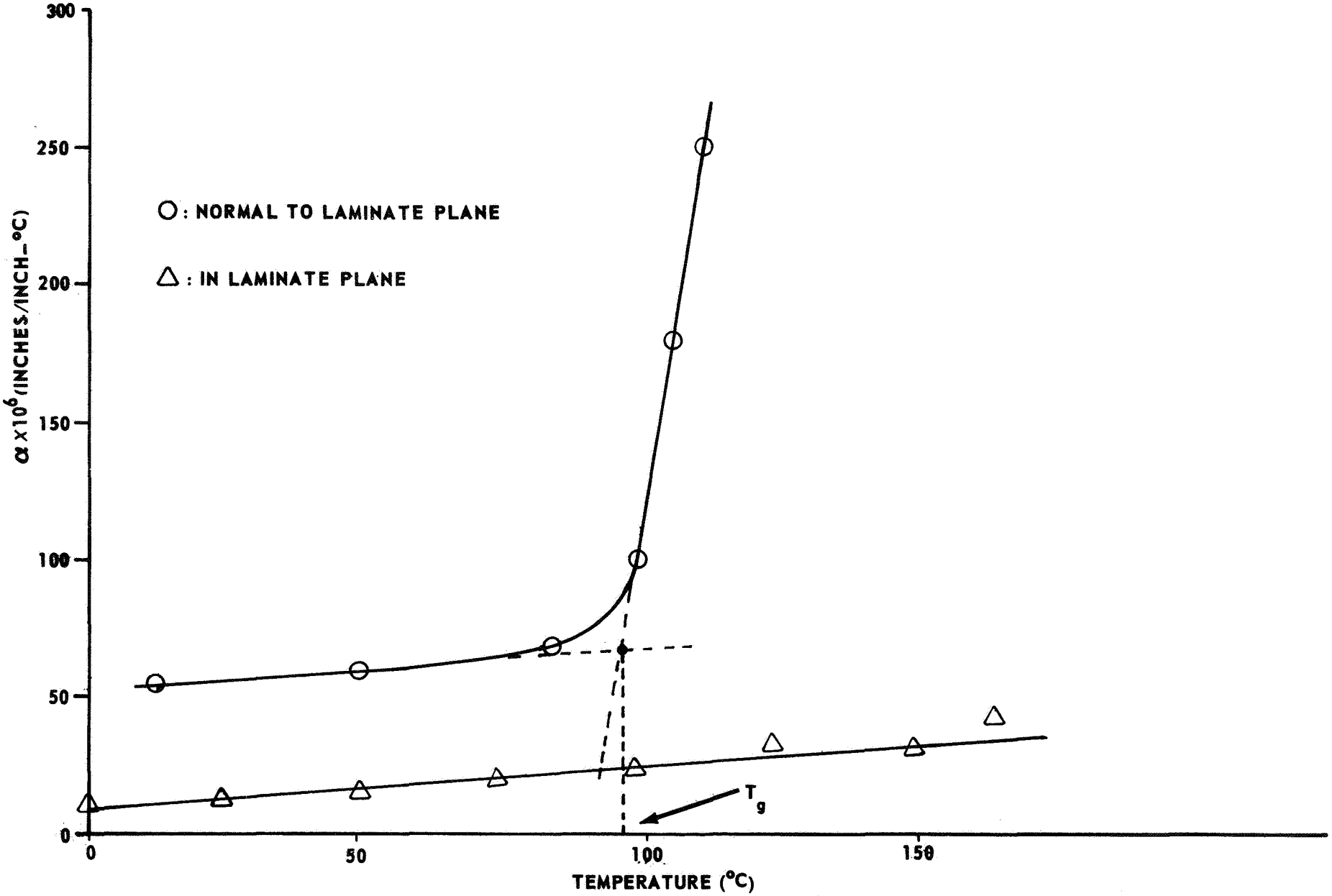


FIGURE 1. COEFFICENT OF THERMAL EXPANSION (α) FOR EPOXY LAMINATE AS A FUNCTION OF TEMPERATURE

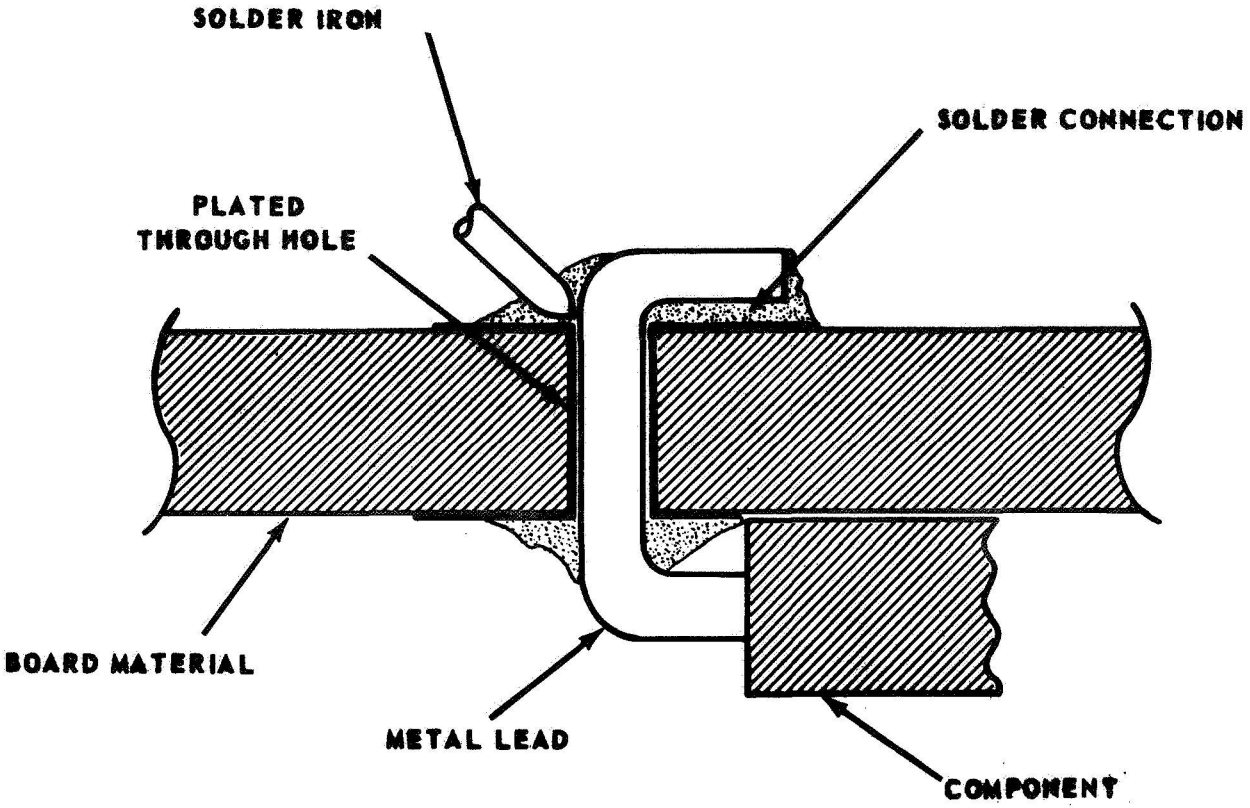


FIGURE 2. PRINTED WIRING BOARD(PWB) SOLDER JOINT

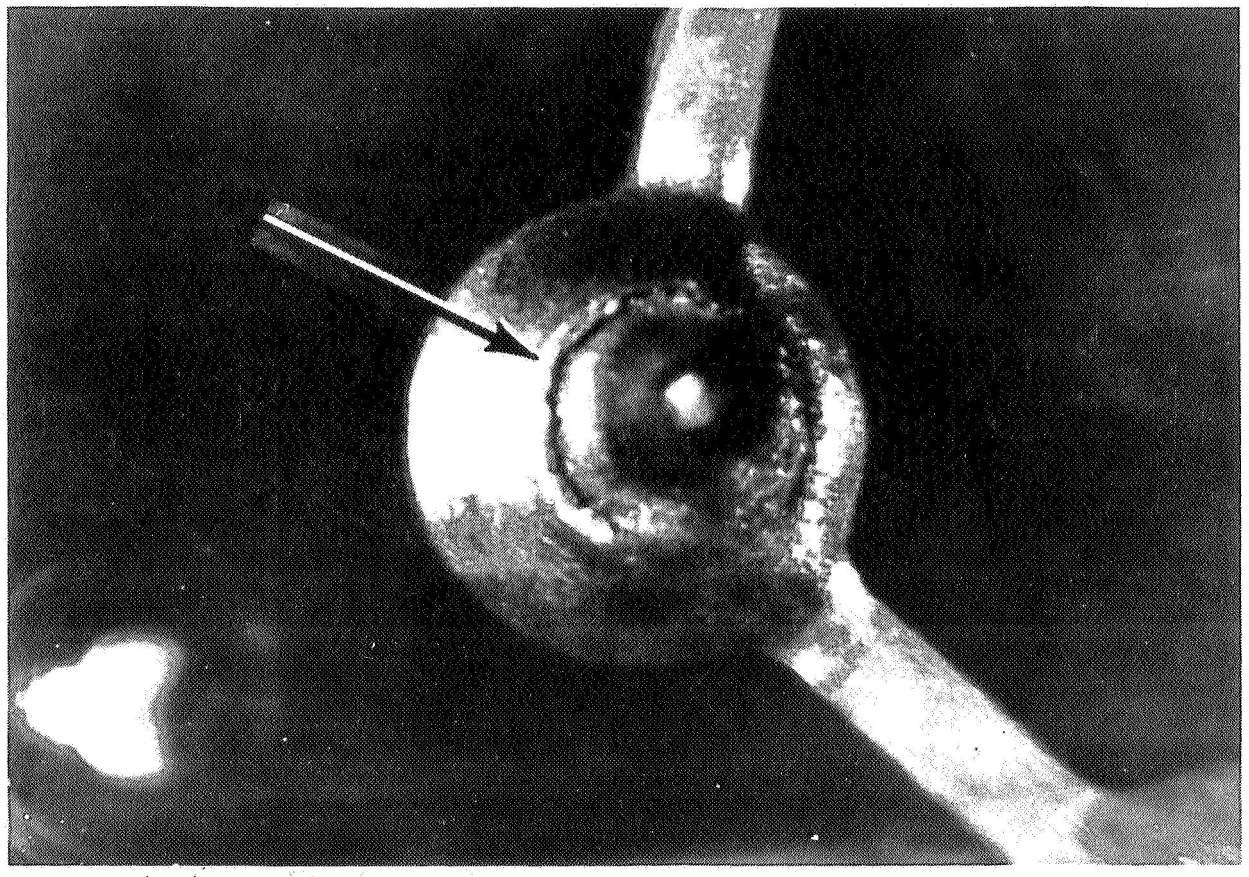


FIGURE 3. CRACKING OF A STRESSED JOINT ON EPOXY PWB DURING THERMAL CYCLING

material represented the logical starting point in development of a minimum-expansion board which was equivalent to G-10 epoxy in other PWB requirements.

TABLE I. EXPANSION OF CIRCUIT BOARD MATERIALS

Materials	Coefficient of Thermal Expansion X 10^6 (in/in-°C)
Epoxy Resin	80-100
30% Epoxy Laminate*	60
Component Leads	15-20
Kovar	5
63/37 Tin/Lead Solder	24
Polyimide Resin	50-55
30% Polyimide Laminate*	45

*30% Resin by weight in laminate; measured below Tg and normal to plane of board.

DEVELOPMENT OF EXPERIMENTAL POLYIMIDE LAMINATE

Materials Section

An effort was initiated to develop a new board design, based on the polyimide material, that would minimize as much as possible the coefficient of thermal expansion (α) normal to the plane of the board. Other basic requirements were also imposed, as shown in Table 2. The need for some sort of particulate filler was anticipated, in order to control the expansion of the laminate. Battelle Memorial Institute provided contractual support in the development of a polyimide-based laminate (Ref. 1). The control of expansion was expected to result from three factors:

(1) Use of a particulate filler with low α-value.

(2) Use of polyimide resin whose α-value is inherently lower than that of epoxy.

(3) Use of a three-dimensionally woven glass cloth reinforcement.

The basic laminate design utilizes three layers of Tricon HS-1, three dimensionally woven fabric as the core of the laminate, with one layer of Style 7628 glass fabric on either side of the three-layer stack to provide an adequate surface finish for the PWB application.

TABLE II. BASIC REQUIREMENTS FOR EXPERIMENTAL POLYIMIDE PWB LAMINATE

Expansion (Normal to Board Plane)	15-20 x 10^{-6} in/in-°C
Thermal Stability	Stable at 200°C
Flammability	Non-Burning (Oxygen-Enriched Environments)
Outgassing	0.2%/cm^2/hour,max. (25°C to 100°C)
Thermal Conductivity	15 x 10^{-4} cal/sec/cm^2/cm/°C,min.
PWB Processing	Equivalent to G-10 Epoxy

Figure 4 illustrates conceptually the laminate composition. The polyimide resin, P-13N, was chosen for its addition crosslinking process, bonding characteristics to the copper foil, and suitability in laminate processing. The P-13N is supplied as a 40 percent solids solution in dimethylformamide in the form of a polyamic acid prepolymer. This type of resin formulation can be terminated with an olefinic reactive monomer such as nadic anhydride. The glass cloth is impregnated with this solution and B-staged to an imidized prepreg, followed by stacking to form the laminate structure and application of heat and pressure. One concept of this sequence in terms of polymer structure is shown in Figure 5. A principal disadvantage of conventional polyimide formulations is the fact that the condensation process resulting in imidization of the polymer is carried out as part of the final laminate-forming step. This results in entrapment of the volatiles from the imidization process in the laminate body and formation of voids. The P-13N system, utilizing addition polymerization processes for further chain extension and/or crosslinking, generates no volatiles and the void content of the laminate is reduced to a minimum.

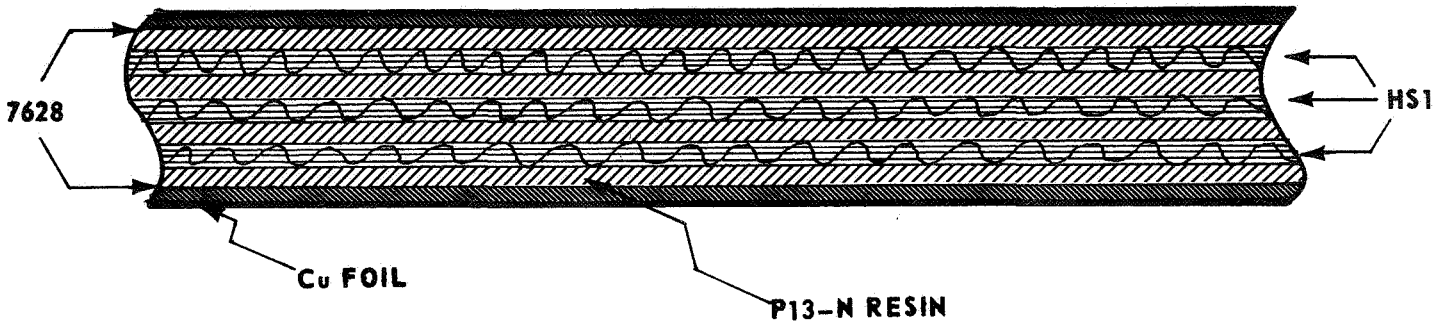


FIGURE 4. CONSTRUCTION OF EXPERIMENTAL POLYIMIDE LAMINATE

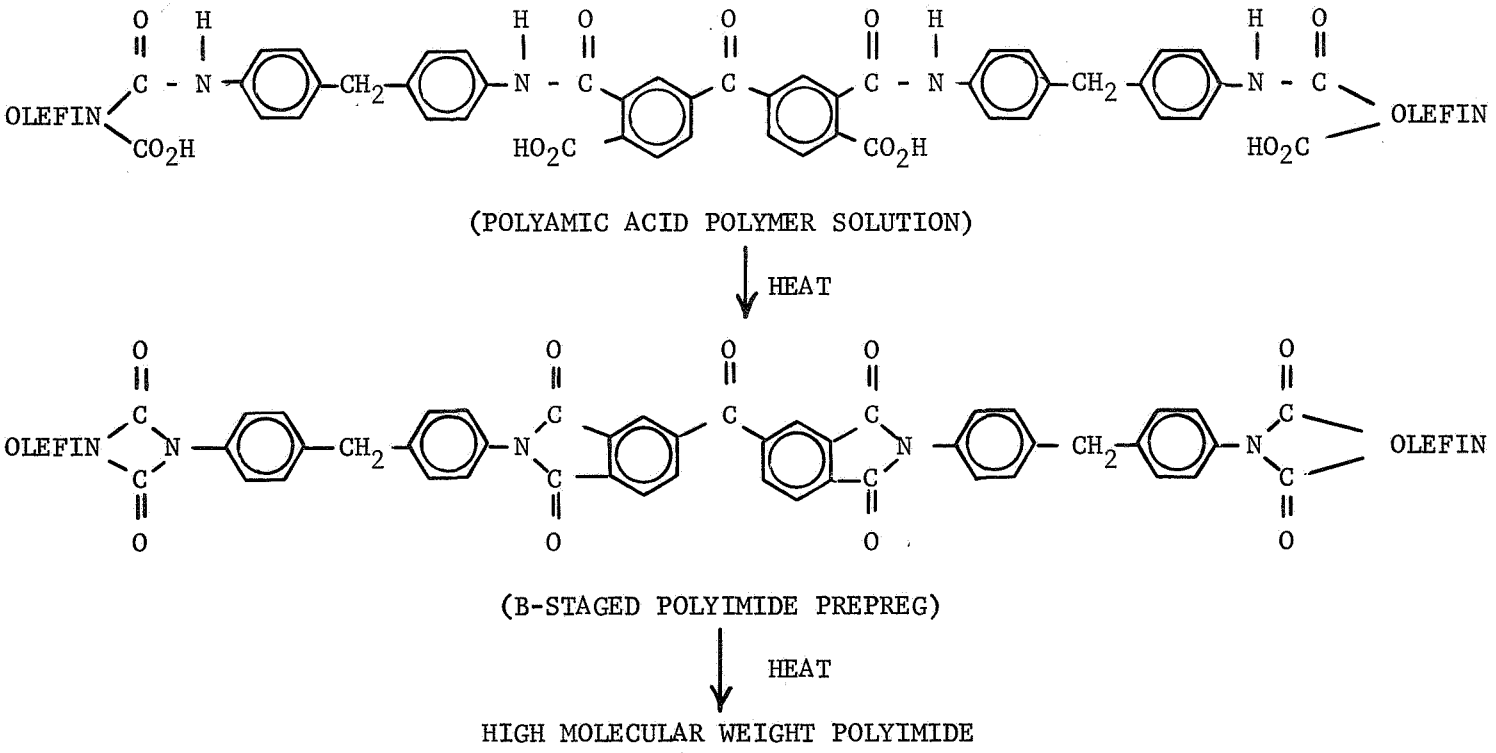


FIGURE 5. POLYIMIDE RESIN PROCESSING SEQUENCE FOR LAMINATE FABRICATION

The particulate filler chosen for this laminate was the A-14 high-fired aluminum oxide, which provided not only control of α normal to the laminate plane but also increased thermal conductivity of the laminate for improved function as a heat sink for heat-generating components. The high-firing treatment of the alumina reduces its capacity for moisture absorption and results in better electrical properties of the laminate. The α-value for this filler is 5-10 x 10^{-6} in/in-°C, so that it is quite effective in counterbalancing the higher expansion of the polyimide matrix.

Laminate Fabrication Process

The optimized laminate design for the experimental polyimide board is shown in Table 3. The fabric impregnation was carried out on a Dixon coater using an impregnating composition of 75.2% resin solution, 24% alumina and 0.77% A-1100 silane coupling agent.

TABLE III. OPTIMIZED POLYIMIDE PWB LAMINATE COMPOSITION

Resin:	P-13N, 40% Solid Solution
Reinforcement:	3 Plies HS-1 Tridirectionally Woven Fabric plus 1 Ply Style 7628 Glass Cloth on either Side of HS-1 Core
Filler:	A-14 High-fired Alumina, 20-22% by Volume
Cladding:	2 oz. Copper with Nickel Strike

This mixture could be successfully homogenized on a ball mill at the 20-22% filler level without subsequent sedimentation. (A complete description of the materials and board fabrication procedure used by Battelle are given in the appendix to this report). The impregnated HS-1 and 7628 fabrics, both with resin/filler contents of 35% by weight, were typically dried 1/2 hour at 150°F (66°C) and B-staged for 1 hour at 400°F (204°C). The prepreg sheets were stacked by orienting each layer at a 90 degree orientation to the adjacent layer. The copper foil was independently coated with P-13N using these same heating conditions.

The stacked plies were typically heated 30 seconds at 600°F (316°C) prior to pressure application. Pressures in the range of 1000-15000 psi

(6,894-10,341 KN/m^2) were applied for 1 hour at the press temperature of 600°F (316°C). The laminate quality was found to be sensitive to the time/temperature/pressure parameters utilized. However, it was found that different time-temperature heating profiles could produce equally acceptable laminates. Also, tests carried out on multiple preparation of laminates by stacking techniques indicate that satif-factory laminates can be prepared in this manner. Thus the board fabrication should be adaptable to commercial practice.

A magnified cross-section of the finished laminate is shown in Figure 6. The opacity of the resin matrix between reinforcing plies in this figure is indicative of the alumina filler loading. The ply count in the figure appears higher than stated due to the weave geometry of the 3 HS-1 plies.

PWB EVALUATION STUDIES

At this point in the development program, two commercially available polyimide-glass laminates coded for purposes of this study as A and B, were included for comparative evaluation. These laminates were obtained as 1 ft^2 sheets having a nominal thickness of 0.062 inch, with 2 oz. copper cladding on both sides. These materials were subjected to the entire evaluation sequence described in this section, along with the experimental polyimide board.

A. Evaluation of Processing Characteristics

1. Drilling Characteristics. This operation was of some concern in the case of the experimental board because of its alumina filler content. Alumina is abrasive and could cause a drill wear problem. The initial drill speed assessed was approximately 22,000 rpm, based on normal drilling operations with the G-10 epoxy board. At this speed, the drill generated dust from drill holes in all the polyimide boards in contrast to the normally acceptable chip formation consistent with smooth hole formation. It was found that a reduction in drill speed to a range of 650-2200 rpm resulted in acceptable "chipping" in all polyimide boards, although 900 rpm appeared to be a lower limit for the experimental board. This determination was made using a #52 carbide drill (0.029 inch diameter). These tests indicated that more drill wear occured on all polyimide boards than on the G-10 epoxy boards, with the experimental polyimide board causing the most significant wear because of the alumina filler.

Additional tests were carried out to determine if the difficulty in drilling the polyimide boards caused disruption of the copper/laminate bond of the copper pads on the drill break-out side of the boards. It

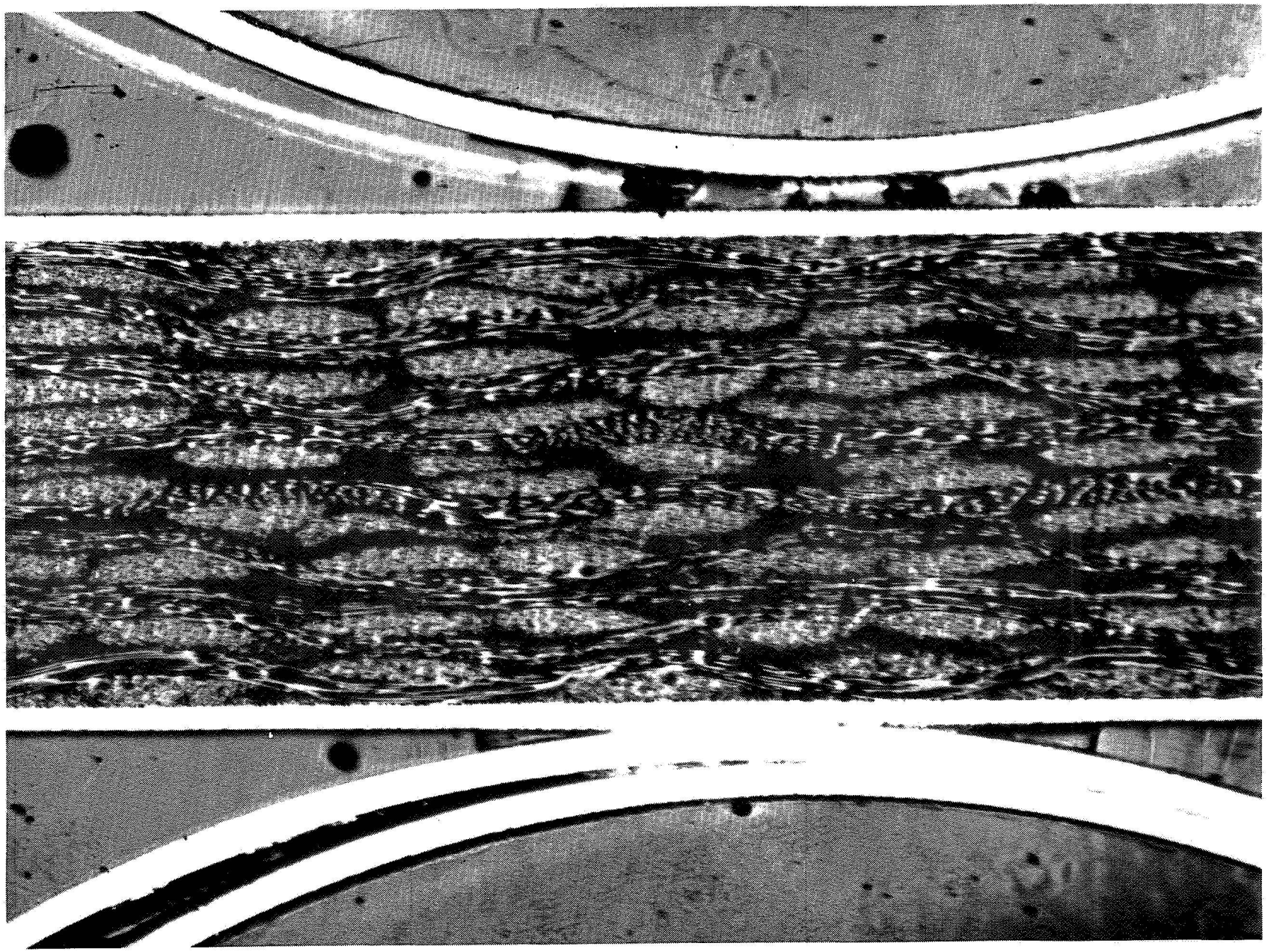


FIGURE 6. SECTION VIEW OF EXPERIMENTAL POLYIMIDE LAMINATES (50X)

was found that when an annular ring of at least 0.010-0.015 inch remained after the hole was drilled, no debond problems occurred and the copper pad/laminate interface was left intact on the break-out side of the board. Figure 7 illustrates the integrity of the copper pad. A specimen of the experimental board was used in this figure, and is typical of the results obtained on the two commercial polyimide boards. The solder pad integrity tests were all performed with an epoxy-glass laminate serving as a backing material on the drill break-out side of the board. Good results were obtained only if a hard sheet such as the epoxy laminate was used.

It was determined through subsequent cleaning and etching exposure tests on the polyimide boards, that the drilling operation should be performed before any copper etching operations. This would preclude any prior weakening of the copper/board adhesive bond.

Examination of the hole walls in the three polyimide specimens revealed a fairly smooth surface, generally equivalent to the G-10 hole wall quality. Sectioned views of the hole walls from these specimens are shown in the scanning electron photomicrographs in Figures 8 and 9. This represents an important consideration for plated through hole applications, where uniform plating thickness on the hole wall and minimum wicking are desired. As shown in Figures 8 and 9, the polyimide PWB hole walls are generally smooth and without any drill marks. Small areas of bare glass fiber reinforcement are observed for each polyimide material. This effect appears less pronounced on the experimental polyimide board specimen. Also as observed in Figure 8, the experimental board shows good integrity of the copper pad/laminate interface both at the drill entry (top of picture) side of the hole, and more critically, at the drill break-out side of the hole.

2. Shearing Characteristics. The commercial polyimides A and B could be sheared on a hand-shearing apparatus using generally the same pressure required for the G-10 laminates, although there was some tendency for B to delaminate during shear. The experimental board required slightly more pressure to shear and the resulting edge appeared rougher than in the case of materials A and B.

3. Etching and Plating Procedures. The polyimide boards generally were processable by the standard etching and plating solutions and procedures utilized in epoxy PWB technology-with one notable exception. The polyimide materials are degraded by strong alkaline solutions. The recurring imide chemical linkage in the polymer backbone is sensitive to base-catalyzed hydrolysis and the hot, basic cleaning solutions normally used in preparing epoxy boards for etching will chemically attack the polyimide boards if the exposure time is sufficient. For this reason, exposure of the polyimide materials to strong basic solutions must be held to an absolute minimum. A typical cleaning and

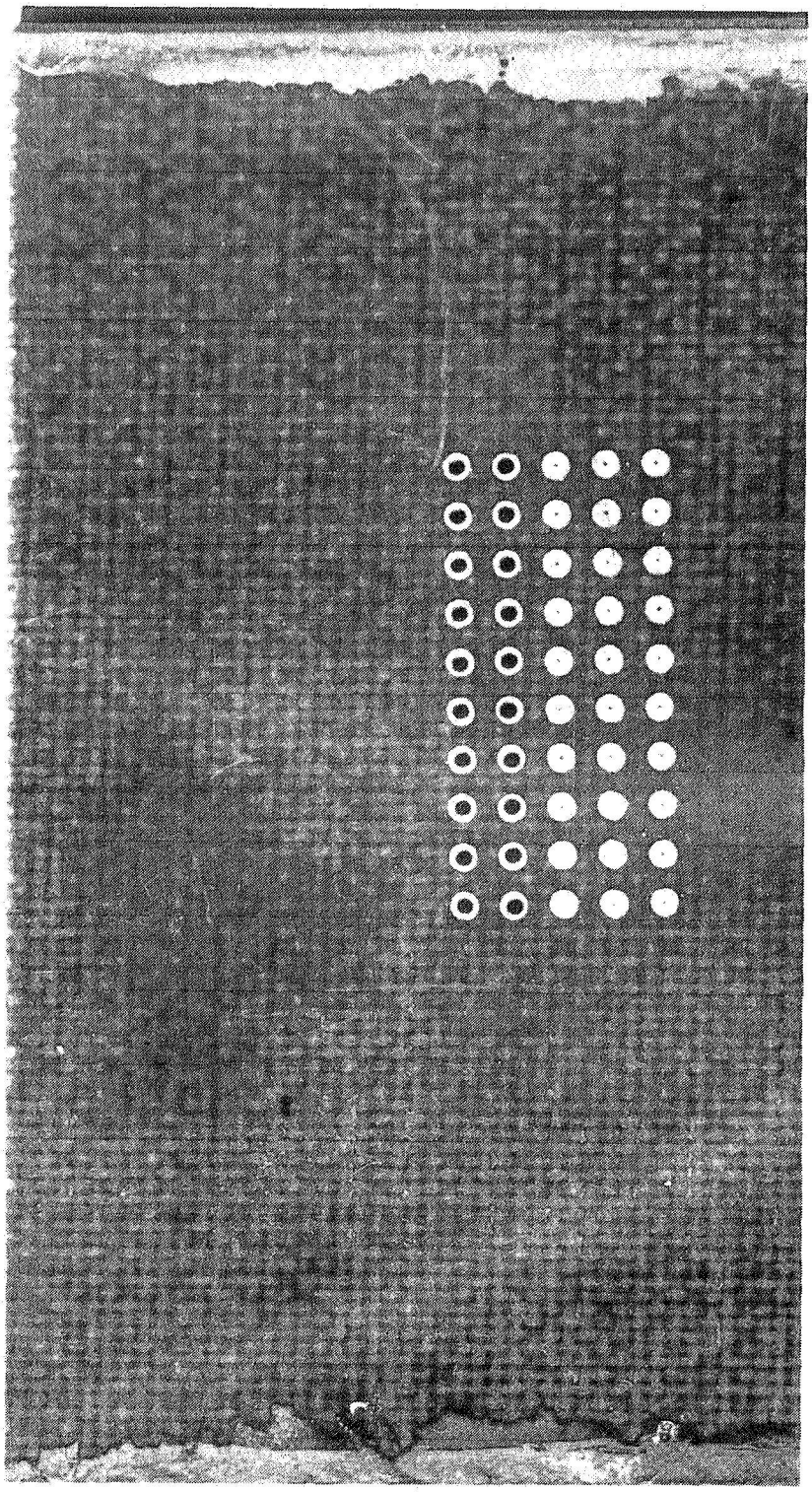
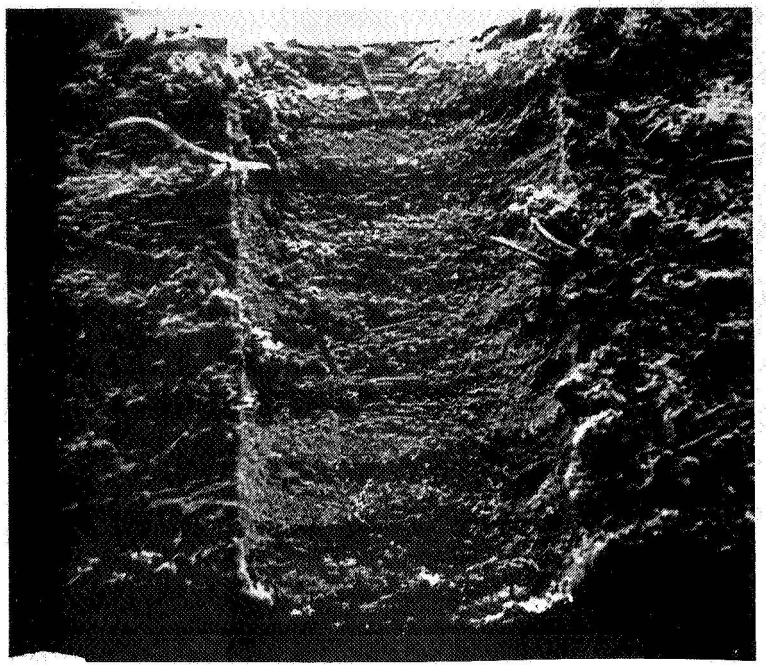
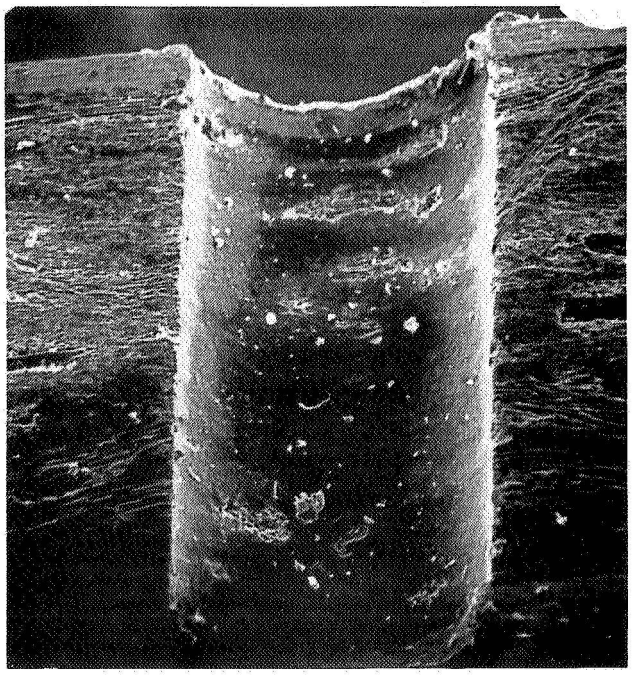


FIGURE 7. SOLDER PAD CONDITION AFTER DRILLING (4X)
(DRILL BREAK-OUT SIDE OF BOARD SHOWN)

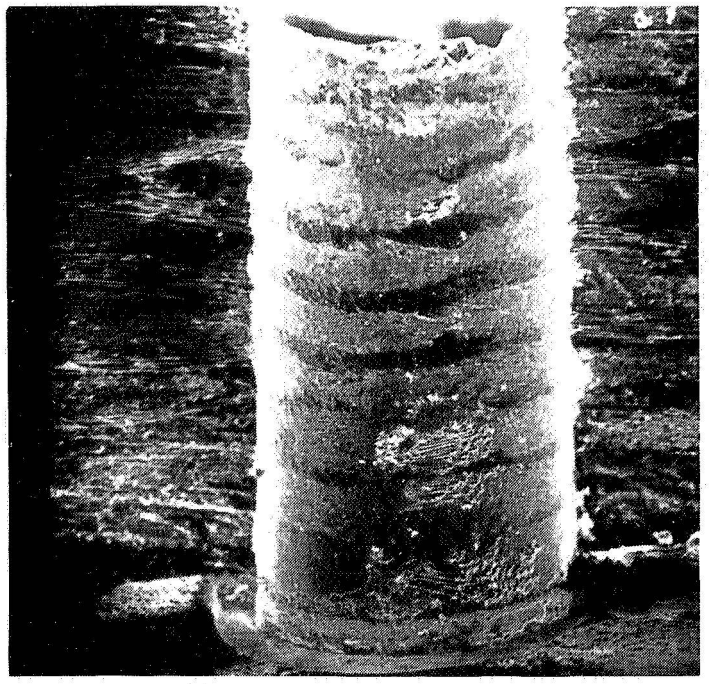


EPOXY PWB

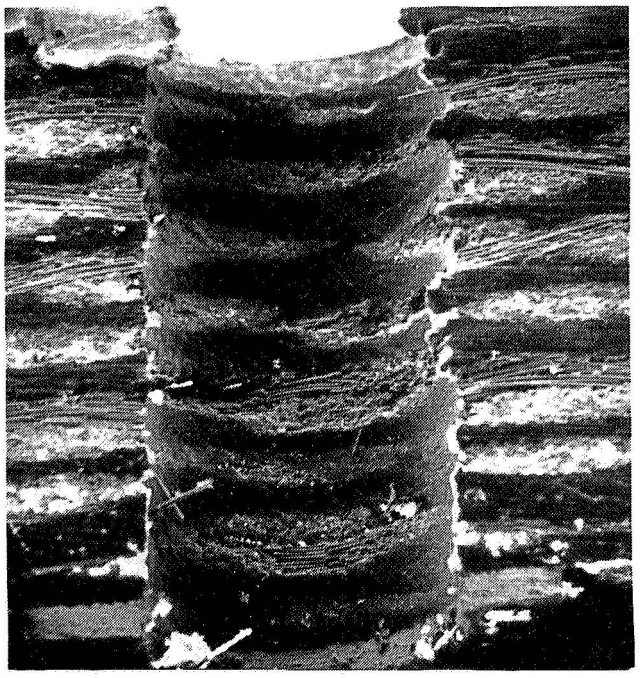


EXPERIMENTAL POLYIMIDE PWB

FIGURE 8. LONGITUDINALLY SECTIONED HOLES FROM EPOXY AND EXPERIMENTAL PWB MATERIALS



PWB A



PWB B

FIGURE 9. LONGITUDINALLY SECTIONED HOLES FROM COMMERCIAL POLYIMIDE PWB A AND B

copper etching sequence for the polyimide boards is given below:

(1) 50% Aqueous NaOH at 50-60°C for 30 seconds.

(2) 25% Aqueous $(NH_4)_2S_2O_8$ at room temperature for 90 seconds.

(3) 25% HCl at room temperature for 30 seconds.

(4) Standard copper etch with $FeCl_3$ at 125-130°F. (52-54°C).

The polyimides show no deterioration under these processing procedures.

The plated through hole processing for these polyimide boards is under investigation and will be discussed in an addendum to this report.

B. Mechanical, Thermal and Electrical Evaluation

The property testing of the candidate boards was carried out in accordance with MIL-P-13949E, "Plastic Sheet, Laminated, Metal-Clad (for Printed Wiring) General Specification for," wherever appropriate, and the testing was on a comparative basis relative to a type G-10 epoxy-glass laminate.

1. Mechanical Property Testing.

a. Copper foil Bond Strength. The adhesive bond strength of the copper foil to the PWB laminate is a critical parameter which was studied in some detail. A standard sequence of environmental exposure tests was carried out in accordance with MIL-P-13949E utilizing the peel test specimen shown in Figure 10. In addition to the initial peel strength values at room temperature, measurements were also made after solder bath exposure, after temperature cycling, at elevated temperature, and after plating solution exposure. These values are summarized in Table 4. The solder exposure consisted of 20 seconds in 260°C liquid solder. The plating solution environment consisted of a sequential exposure in sodium hydroxide, sodium cyanide, and sulfuric acid as defined in MIL-P-13949E. The temperature cycling conditions involved 5 cycles from -65°C to +125°C.

The copper/board bond strength was also assessed by a type of terminal pull test, wherein specimens such as that shown in Figure 11 were etched to provide a series of solder pods. Buss wire leads, 22 gauge, were soldered to one side of the board using soldering procedures in MSFC-STD-154. The leads were then subjected to a tensile force normal to the plane of the board with an Instron tester using a crosshead speed of 0.5 inches/minute. The temperature was varied from room

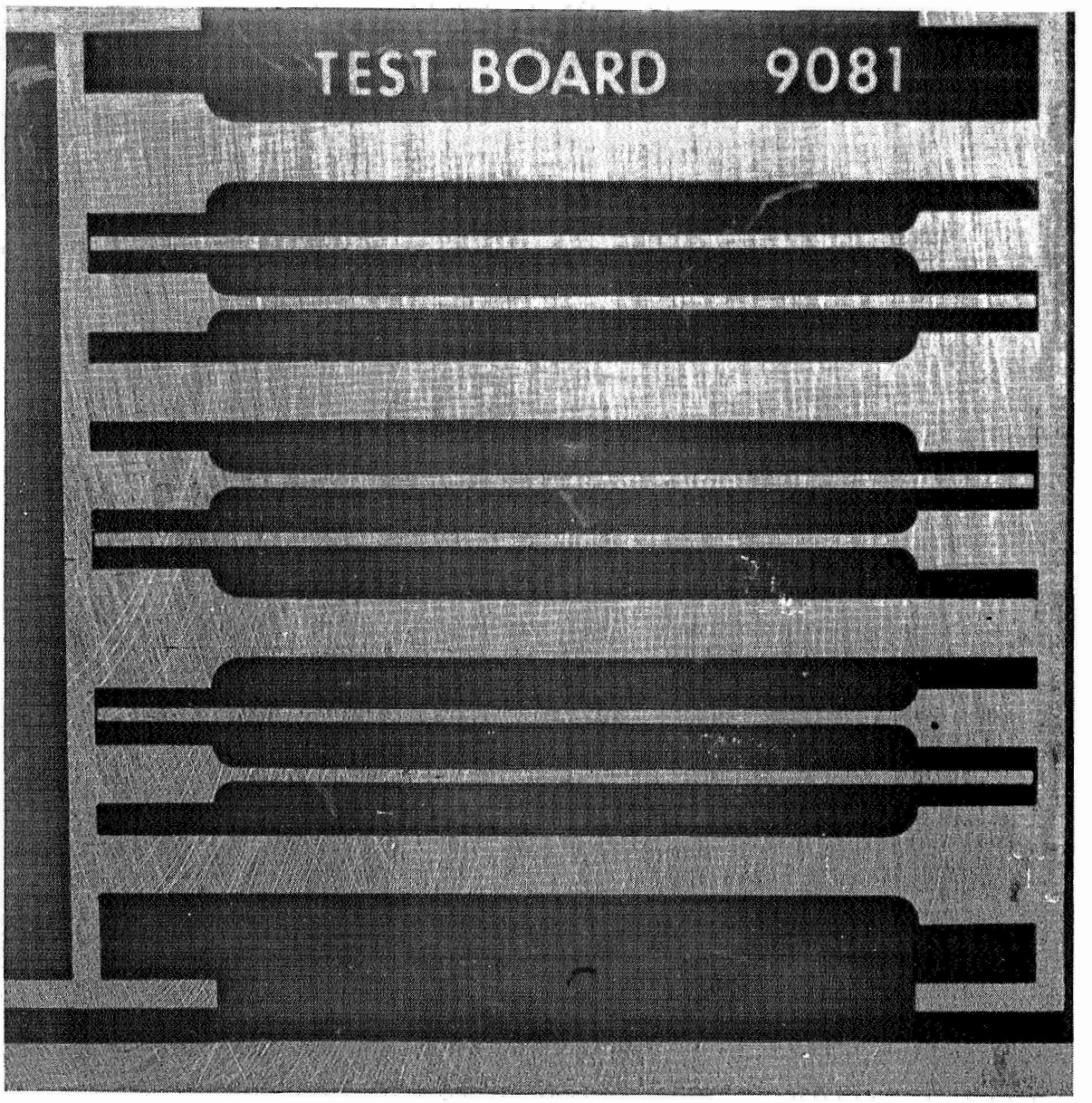


FIGURE 10. COPPER FOIL PEEL STRENGTH TEST SPECIMEN

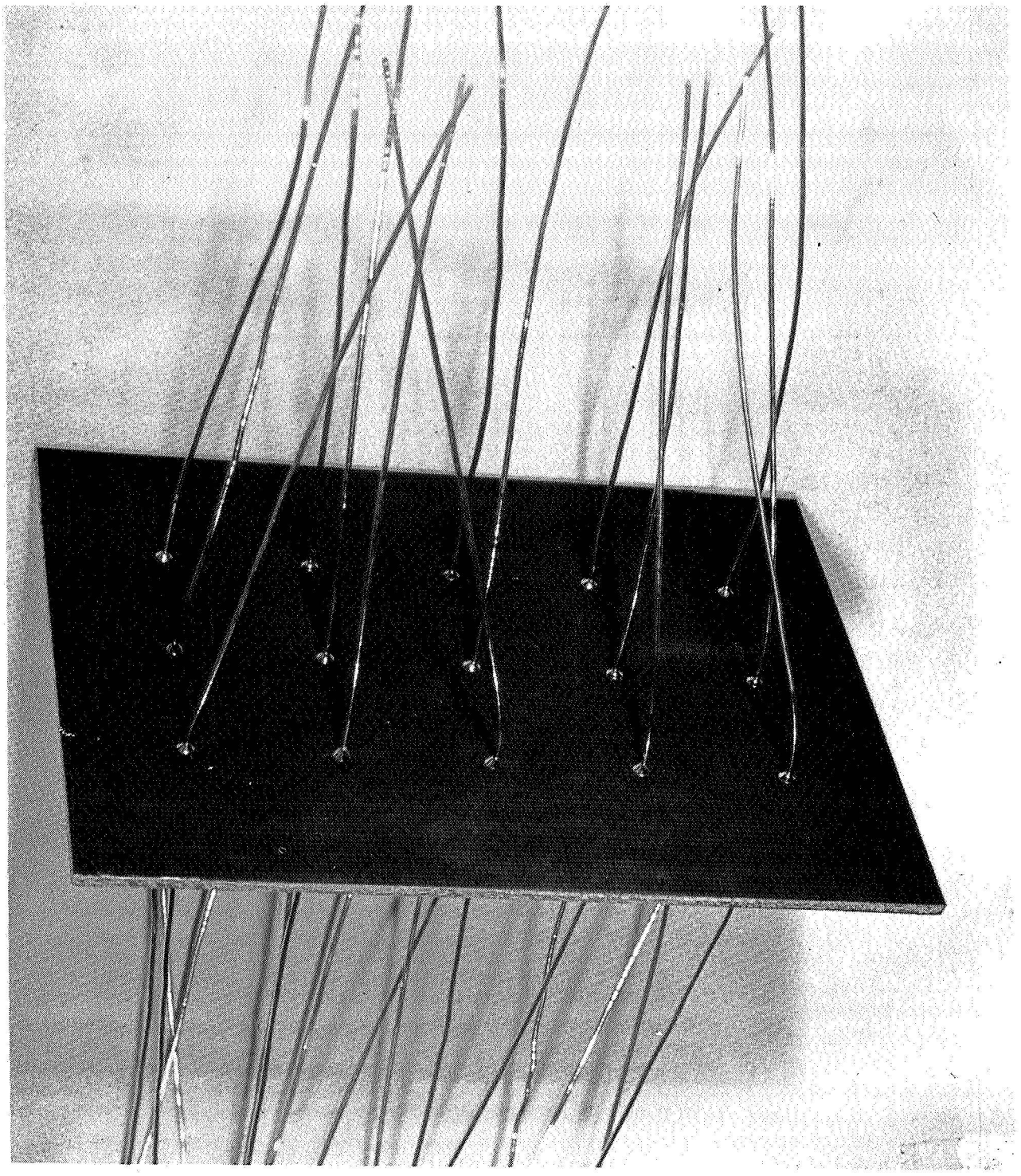


FIGURE 11. TERMINAL PULL SPECIMEN

temperature to the melting point of 60/40 tin/lead solder, approximately 188°C. The tensile force required to pull the solder joint off the boards as a function of temperature is summarized in Figure 12.

TABLE IV. PEEL STRENGTH OF COPPER FOIL ON PWB SUBSTRATES

Condition	Experimental Board		Board A		Board B		Epoxy Board	
Control:	2.03	(11.4)	2.12	(11.9)	2.54	(14.3)	2.65	(14.9)
After Solder Dip:	1.64	(9.2)	2.03	(11.4)	1.83	(10.3)	2.19	(12.3)
After Plating Solution Exposure:	1.71	(9.6)	2.01	(11.3)	2.60	(14.6)	2.06	(11.6)
After Temperature Cycling:	1.62	(9.1)	2.31	(13.0)	2.06	(11.6)	1.89	(10.6)
After 250°C Exposure:	1.49	(8.4)	2.05	(11.5)	2.54	(14.3)	1.42	(8.0)
At 150°C:	1.71	(9.6)	1.57	(8.8)	1.99	(11.2)	0.73	(4.1)
At 205°C:	1.49	(8.4)	1.42	(8.0)	1.89	(10.6)	0	
At 260°C*:	1.07	(6.0)	1.07	(6.0)	1.07	(6.0)	0	

(All peel strengths given in KN/M of width; pounds/inch in parentheses)
* Copper strip underwent tensile failure

b. Flexural Strength. The flexural strength of the candidate materials was measured as a function of temperature from room temperature to 260°C in order to quantify the improved dimensional stability and strength retention of the polyimide laminates at elevated temperatures. The tests were performed in accordance with ASTM D790, using specimens having dimensions of 3.0 inches (7.62 cm.) long by 0.042 inches (1.07 cm.) wide. The flexural strength was determined at room temperature, 149°C, 204°C, and 260°C. The results are plotted in Figure 13, with flexural strength in units of kilonewtons/meter^2.

The candidate boards were also thermally cycled to determine if any loss of flexural strength occurred. The materials were cycled

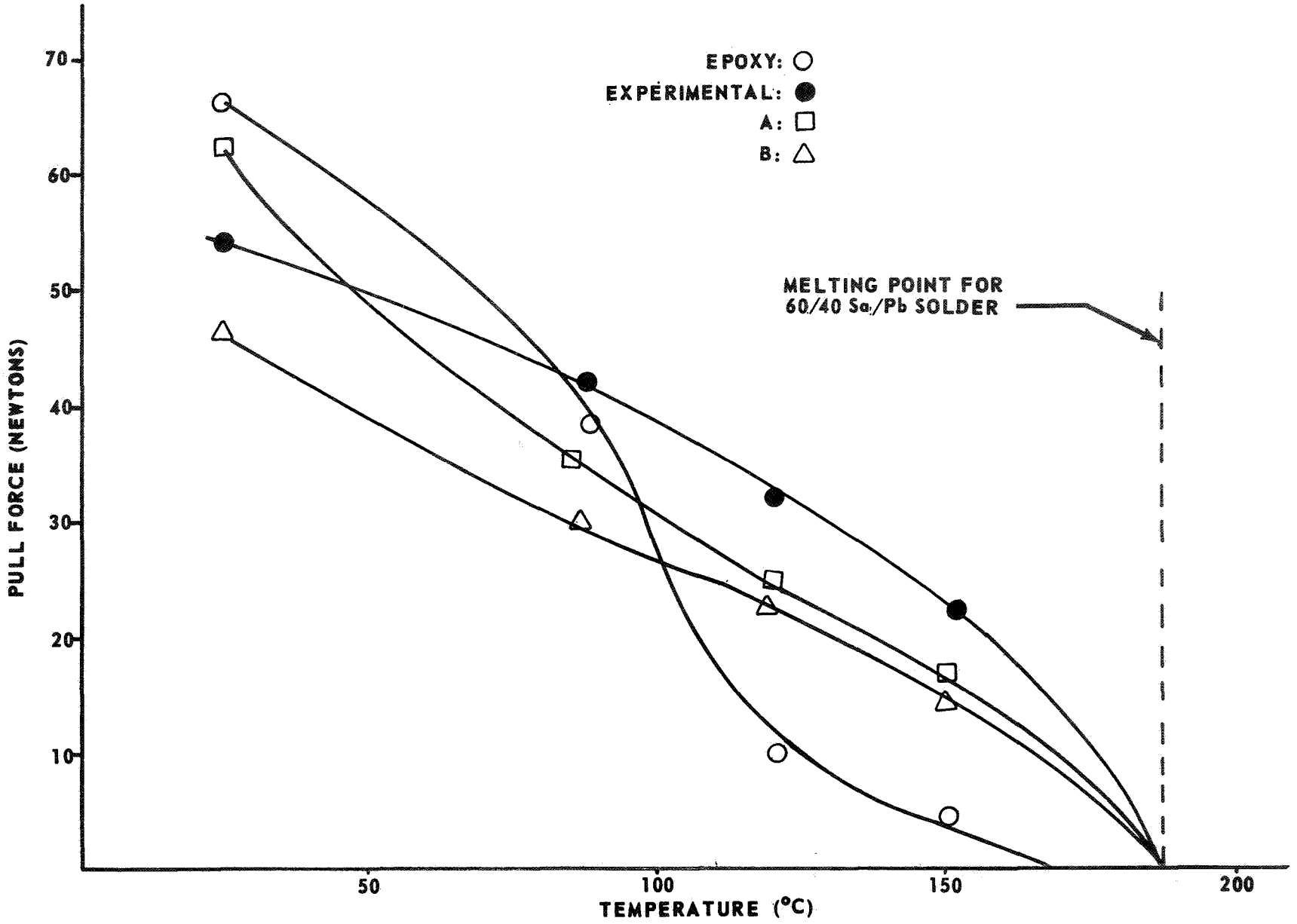


FIGURE 12. TERMINAL BOND STRENGTH VS. TEMPERATURE FOR POLYIMIDE LAMINATES

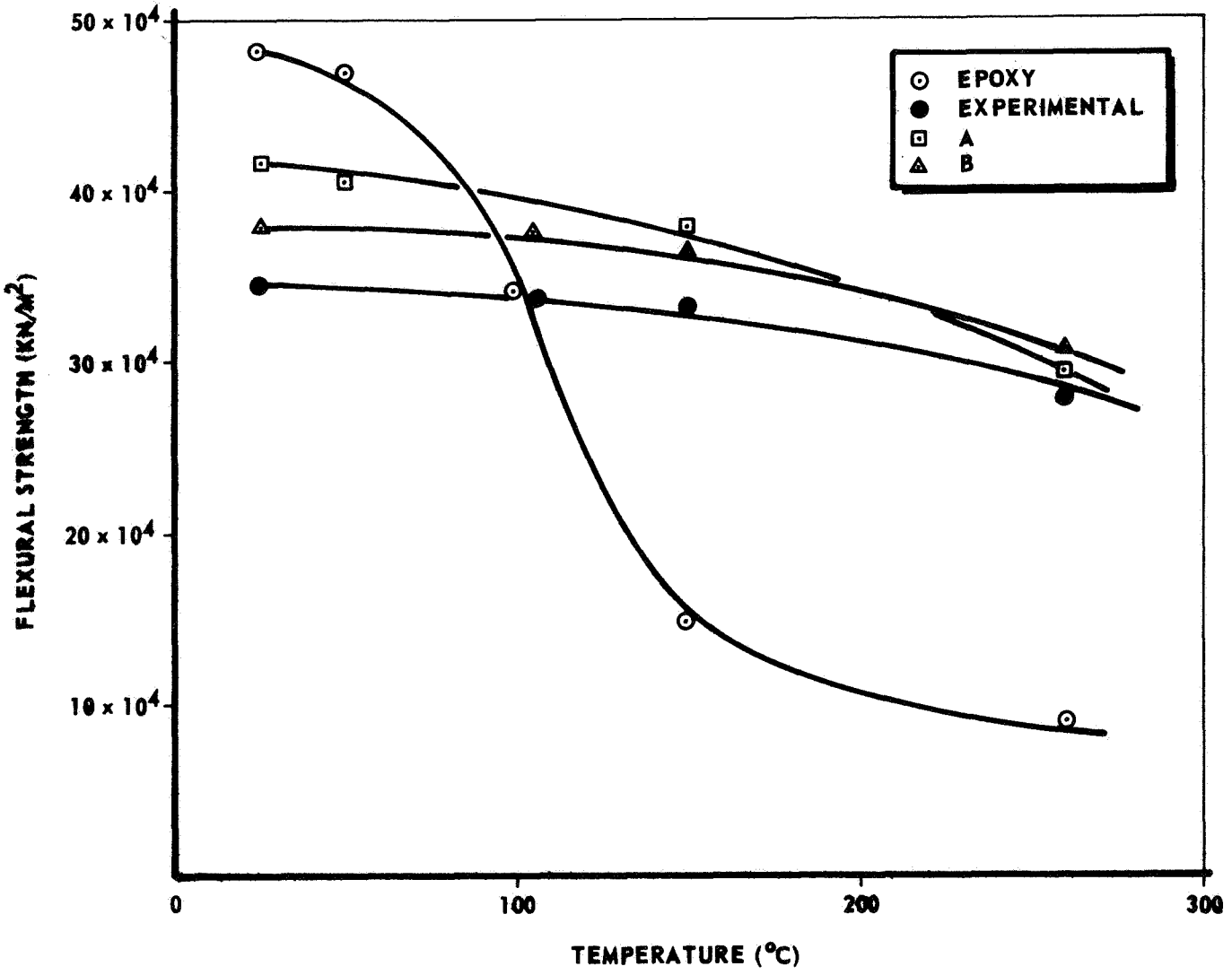


FIGURE 13. FLEXURAL STRENGTH vs. TEMPERATURE FOR POLYIMIDE LAMINATES

four times every 24 hours from -55°C to +100°C for a total cycle time of 14 days. Microscopic examination of all specimens did not reveal any delamination or other evidence of deterioration. No significant change in room temperature flexural strength was found following this conditioning.

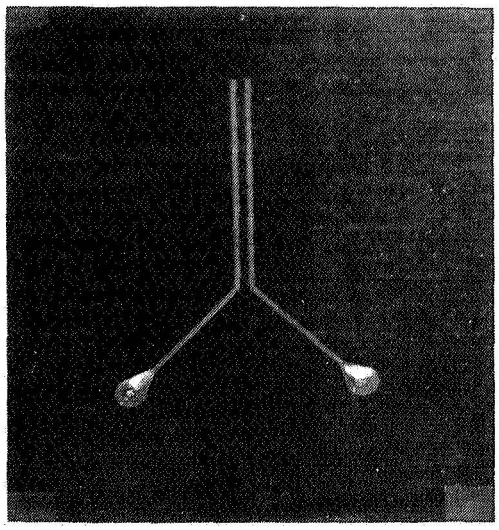
2. Electrical Property Evaluation. A series of electrical property tests was performed on the boards to determine if they provided an adequately insulating substrate for printed wiring conductors.

a. Insulation Resistance. The insulation resistance of the candidate boards was measured in accordance with the requirements of MIL-STD-202D, Method 302, Condition B, using a Keithley Model 515 megohm bridge. The test conditions were 500 volts DC applied for 1 minute. The test specimens were standard Y-pattern test boards described in MIL-P-13949E, and shown in Figure 14. Three specimens of each polyimide type were measured and the value reported is an average from the three specimens. Measurements were made at room temperature, at 125°C, at room temperature following the 125°C exposure, and after temperature cycling at 95% relative humidity. The data are summarized in Table 5.

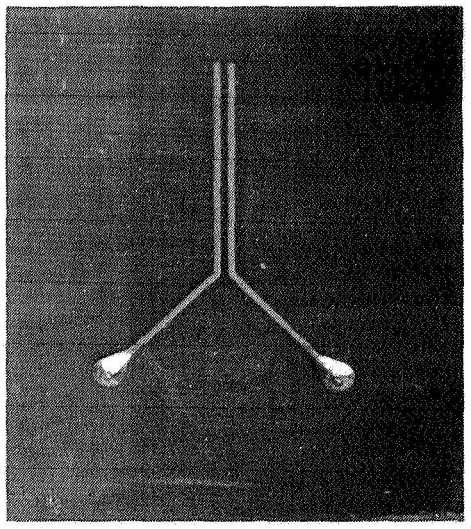
TABLE V. INSULATION RESISTANCE OF PWB MATERIALS

Board Material	I.R. (OHMS) Room Temperature	I.R. (OHMS) At 125°C	I.R. (OHMS) After Humidity Cycling	I.R. (OHMS) After 125°C Exposure
Epoxy	45×10^{12}	14×10^{9}	5×10^{10}	1×10^{12}
PWB A	55×10^{12}	12×10^{9}	15×10^{12}	50×10^{12}
PWB A	60×10^{12}	5×10^{9}	50×10^{11}	20×10^{12}
Experimental PWB	75×10^{12}	12×10^{9}	14×10^{12}	60×10^{12}

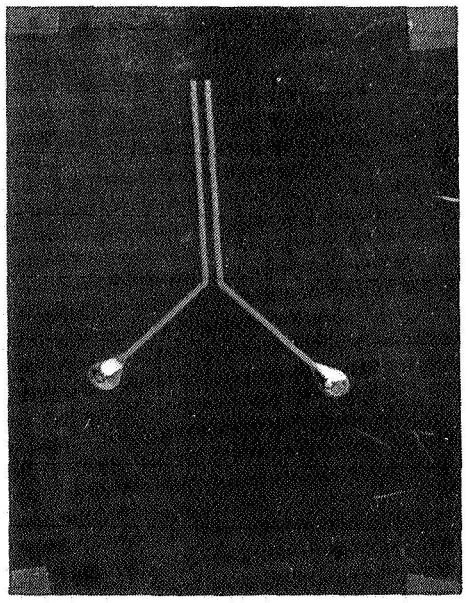
The same boards were used throughout the test sequence, so that any degradation due to the test environments would be cumulative. Following each of the last two exposure periods in Table 5 the test boards were allowed to return to ambient conditions and stabilize for 1 hour before any insulation resistance measurement was made. For the test at 125°C, the boards were stabilized for 1 hour at that temperature



EXPERIMENTAL PWB



PWB A



PWB B

FIGURE 14. POLYIMIDE ELECTRICAL TEST SPECIMENS

before a measurement was made. The temperature cycling exposure at 95% relative humidity consisted of 8 cycles, each of which was 24 hours in duration and composed as follows:

25°C to 71°C in 2 hours

Hold at 71°C for 6 hours

71°C to 25°C in 16 hours

b. Dielectric Constant and Dissipation Factor. These measurements were carried out initially at 1MHz using a General Radio Model 716-CSI capacitance bridge and model 1690 dielectric sample holder. Circular PWB specimens 2 inches (5.08cm) in diameter were utilized with the 0.002 inch (0.00508 cm) copper foil on each side to serve as an intimate contact electrode. Table 6 shows these data at 1 MHz.

TABLE VI. DIELECTRIC CONSTANT AND DISSIPATION FACTOR FOR PWB LAMINATES

Board	D. C. at 1 MHz	D. F. at 1 MHz
Epoxy	4.8	0.019
Experimental PWB	5.2	0.004
PWB A	4.7	0.006
PWB B	4.3	0.005

These two dielectric properties were then measured as a function of frequency and temperature as summarized in Figures 15 and 16. Measurements were made over a frequency range of 10 to 10^6 Hz using a General Radio Model 716C capacitance bridge, and over a temperature range of 25-175°C utilizing an environmental chamber.

c. Volume and Surface Resistivity. These measurements were made in accordance with the test and specimen configuration described in MIL-P-13949E. The guarded electrode specimen was prepared by etching the copper foil as illustrated in Figure 17. The copper foil geometry on the opposite side of the specimen in Figure 17 was a solid disc having an idential diameter as the outer diameter of the concentric ring shown. The test was performed with a Keithley Model 515 megohm bridge with a Keithley Model 6105 resistivity adapter as the test

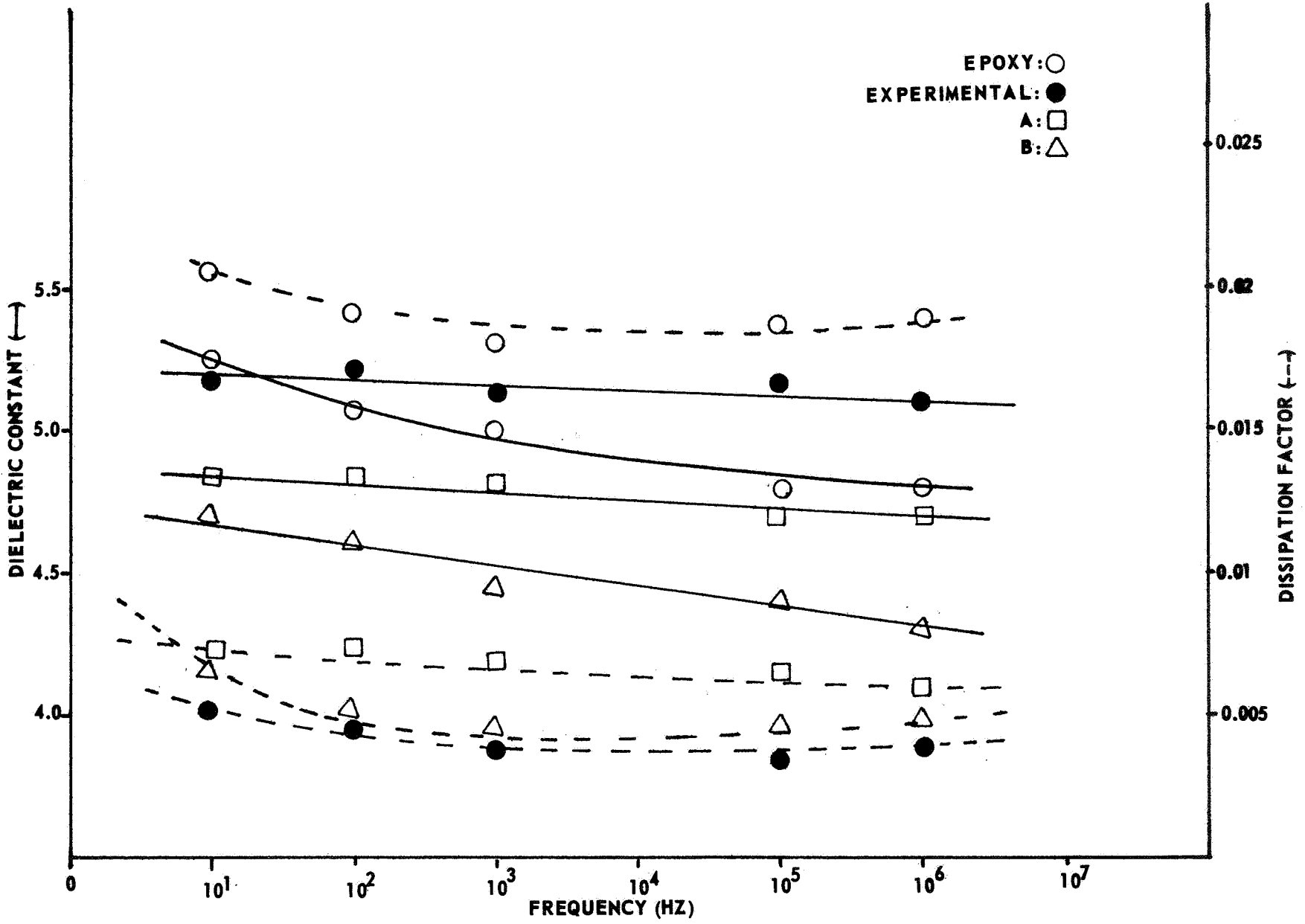


FIGURE 15. DIELECTRIC CONSTANT AND DISSIPATION FACTOR VS. FREQUENCY FOR POLYIMIDE PWB

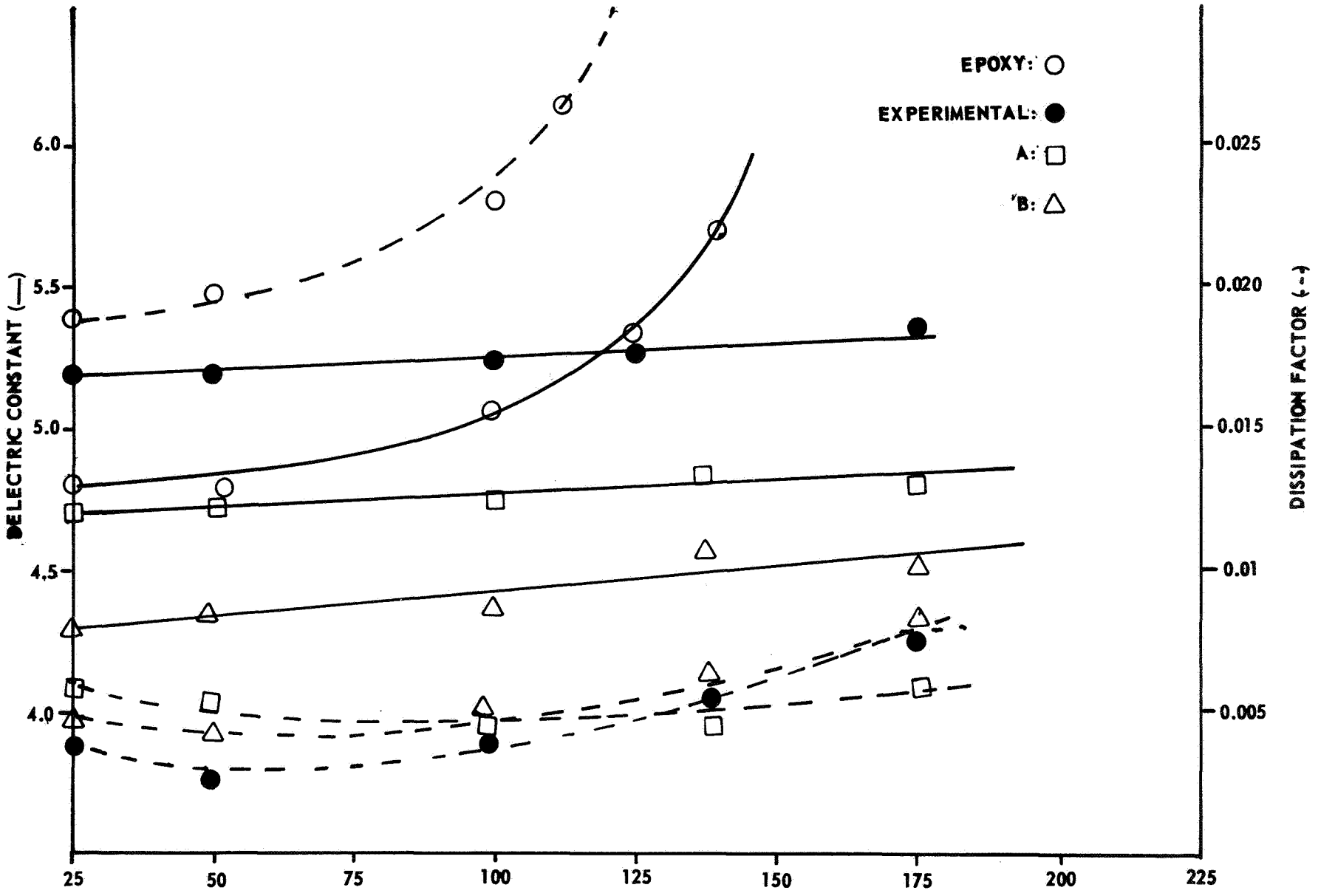


FIGURE 16. DIELECTRIC CONSTANT AND DISSIPATION FACTOR VS. TEMPERATURE FOR POLYIMIDE PWB

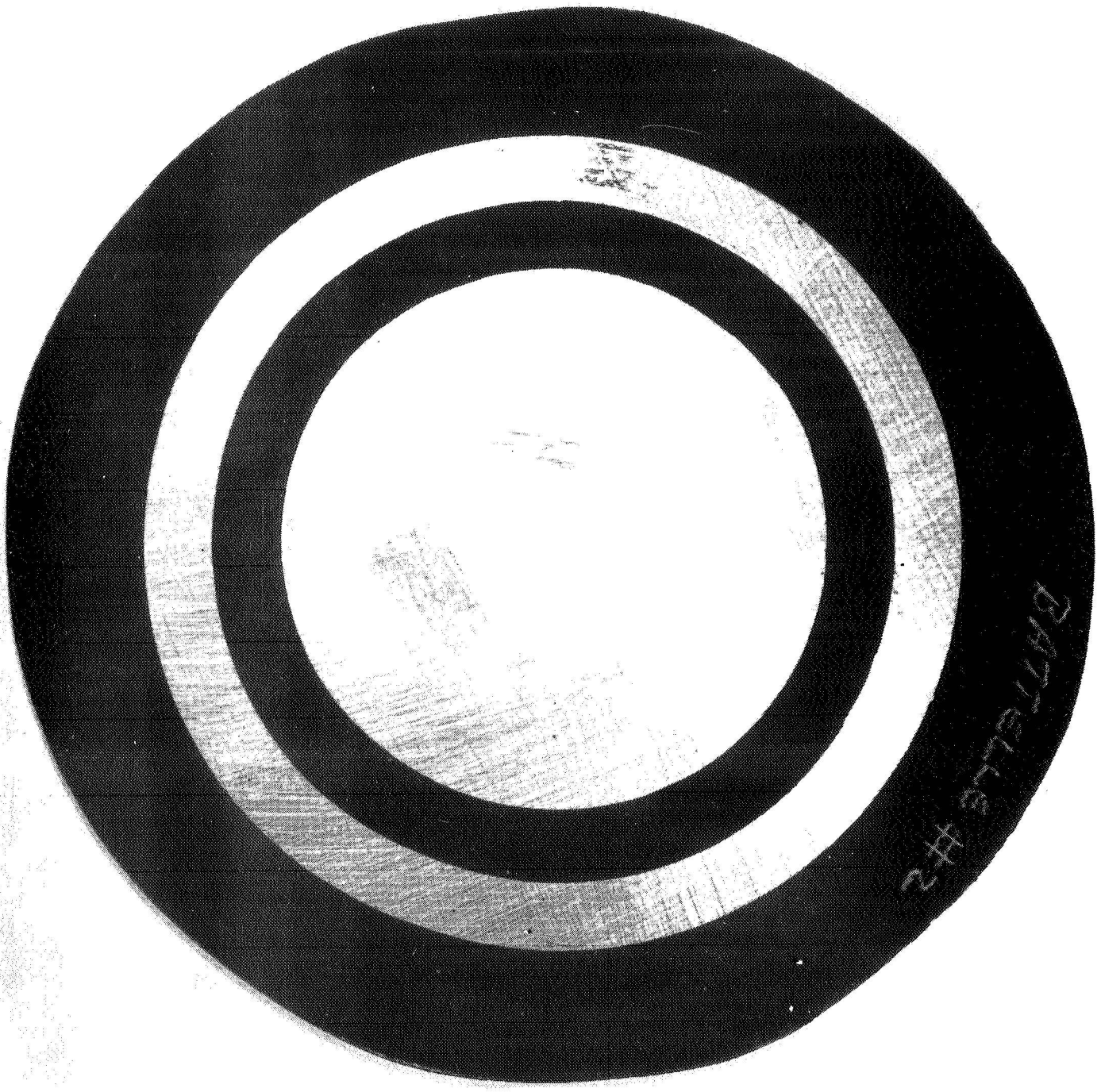


FIGURE 17. POLYIMIDE PWB RESISTIVITY TEST SPECIMEN

fixture. The data obtained for the PWB specimens are presented in Table 7.

TABLE VII. VOLUME AND SURFACE RESISTIVITY FOR POLYIMIDE PWB MATERIALS

Board	Volume Resistivity (Ohm-Cm)	Surface Resistivity (Ohms)
Epoxy	9.5×10^{13}	1.70×10^{13}
Experimental	1.16×10^{14}	1.65×10^{13}
A	1.15×10^{14}	1.56×10^{13}
B	1.13×10^{14}	1.40×10^{13}

d. Dielectric withstanding Voltage. This test was performed in accordance with MIL-STD-202D, Method 1, to assess the ability of the polyimide materials to function acceptably as dielectrics under momentary surges of voltage without appreciable leakage current. The test equipment consisted of a Peschel Model P1-10X voltage source, equipped with an automatic voltage rate-of-rise control and a microammeter for measuring leakage current. The Y-pattern test boards were used for this test. The test board leads were connected to the instrument and 1000 volts AC were applied at the rate of 500 volts/second. The test voltage was held at 1000 volts for 1 minute while monitoring the micrommeter for any leakage current. Also the conductor traces on the test board were observed closely for any evidence of air discharge or arcing. The test was repeated after 1 hour exposure of the same test boards at 100°C and again after the boards were temperature cycled for 5 days at 95% relative humidity, using the cycle described earlier for the insulation resistance testing. The results are given in Table 8. The boards were allowed to stabilize for 1 hour following each environmental exposure test before the dielectric withstanding voltage test was performed.

TABLE VIII. DIELECTRIC WITHSTANDING VOLTAGE TEST ON POLYIMIDE PWB MATERIALS

Board	Leakage Current (Microamperes) Initial	After Temp. Exposure	After Temp./Hum. Exposure
Epoxy	2.3	1.1	2.8
Experimental	2.0	1.3	2.9
A	2.5	1.3	2.8
B	2.1	1.0	2.8

e. Arc Resistance. The arc resistance testing was carried out on the polyimides to determine their ability to resist formation of a conducting path when a high voltage arc was applied at the surface of the specimen. The copper foil was removed from specimens 5 inches in diameter and the test was performed as specified in MIL-P-13949E using an Associated Research Model 8540 Tester capable of generating 12.5 kilovolts. The data obtained from this test reflected the time-to-failure of the specimen, where failure was defined as generation of a continuous arc (between the two instrument electrodes) which subsequently disappears into the material. These data are presented in Table 9.

TABLE IX. ARC RESISTANCE TEST DATA FOR POLYIMIDE PWB MATERIALS

Board	Time to Failure (Sec)
Epoxy	180.2
Experimental	185.3
A	184.3
B	182.4

f. Dielectric Breakdown. This test was carried out in accordance with MIL-P-13949E. The equipment consisted of an Associated Research Model 4510-M3 AC high potential tester with appropriate specimen fixtures. The testing was performed with the voltage applied both parallel to the plane of the board as shown in Figure 18 and normal to the plane of the board by placing 1 inch diameter electrodes in opposing positions above and below the board. The specimens shown in Figure 18 were prepared by drilling two 0.1875 inch (0.476 cm) holes that were 1 inch (2.54 cm) apart, center-to-center. The 3.0 inch (7.62 cm) tapered electrodes were inserted until they extended through the PWB specimen to an equal length on each side. The test fixture was submerged in high dielectric strength silicone oil to prevent flash-over between electrodes. In both specimen configurations the voltage was applied at the rate of 500 volts/second and continued until either the specimen failed or the instrument limit of 50 KV was reached. In the case of the tapered pin electrode configuration (Figure 18) the instrument limit was reached without failure for each polyimide as well as the epoxy. In the opposing electrode configuration, however, the electrodes are separated only by the thickness of the board specimen (nominally 0.062 inch, 0.157 cm.) and dielectric breakdown was observed. Table 10 summarizes these data for the test boards.

TABLE X. DIELECTRIC BREAKDOWN TEST DATA FOR POLYIMIDE PWB MATERIALS

Board	Breakdown Voltage Parallel to Board (KV)	Breakdown Voltage Perpendicular To Board (KV)	Dielectric Strength (Volts/Mil)
Epoxy	50	38.25	659
Experimental	50	34.18	600
A	50	36.94	616
B	50	43.46	712

The numerical values of breakdown voltage were obtained by a stepwise voltage application, whereby the voltage was increased from zero in 2.5 KV increments until failure occurred. The values of dielectric strength in Table 10 are obtained by dividing the breakdown voltage by the specimen thickness, and this parameter is conventionally expressed as volts/mil.

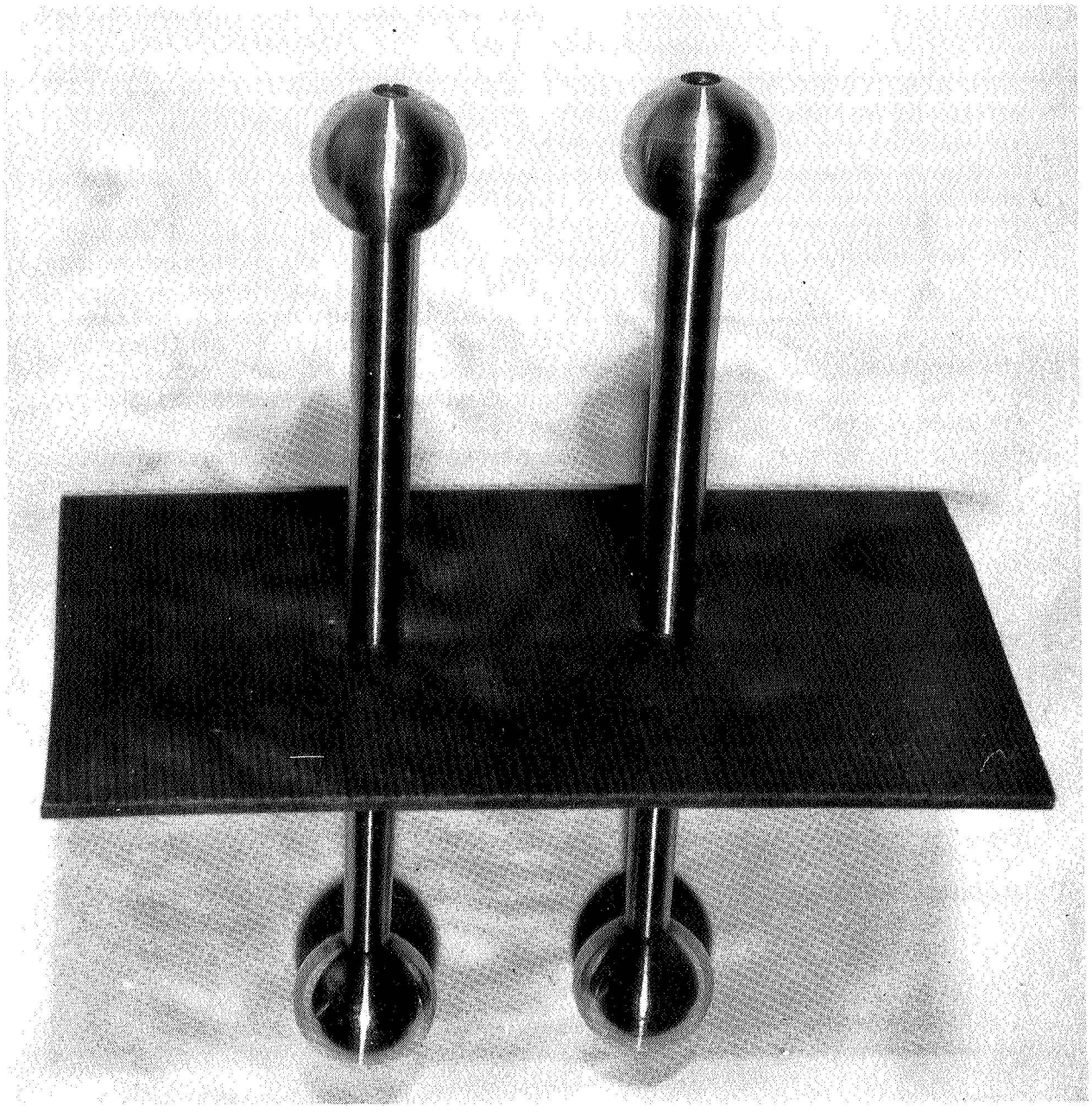


FIGURE 18. DIELECTRIC BREAKDOWN TEST SPECIMEN FOR POLYIMIDE PWB MATERIALS

3. Thermal/Physical Property Evaluation. The tests described in this section deal primarily with the thermal characteristics of the polyimide PWB materials. Other physical property tests not appropriate to the preceeding sections are described also.

a. Linear Coefficient of Thermal Expansion (α). The evaluation of the thermal expansion characteristics was performed on a Perkin-Elmer Model TMS-1 thermomechanical analyzer with a Perkin-Elmer Model UU-1 temperature programming controller and a Hewlett-Packard Model 7001-AR X-Y recorder. The analyzer operates by utilizing a linear variable differential transformer which serves as a displacement transducer to sense changes in dimension of the sample and to convert them to electrical signal for the X-Y recorder.

The critical axis with respect to the PWB expansion characteristics is normal to the plane of the board and the test specimens were prepared as discs, 0.250 inches (0.635 cm) in diameter, and having the normal thickness of the PWB sheet from which they were cut. This thickness was typically 0.58 - 0.62 inch (0.147 - 0.157 cm). The test equipment is sensitive to 5×10^{-6} inches/inch and is reproducible on a given sample to within $\pm 1 \times 10^{-6}$ inches/inch-°C in measurement of coefficient of expansion (α). The instrument was programmed in the expansion mode at the rate of 10°C/minute over the temperature range of -100°C to +250°C. The instrument output is recorded automatically on the X-Y recorder as displacement versus temperature. The most critical temperature region was -55°C to +100°C corresponding to the thermal cycling conditions for the test boards discussed in the next section. Thus the expansion coefficient (α) for each PWB material represents an average from -55°C to +100°C. For comparative purposes α-values over higher temperature ranges were also calculated, and all experimental data are summarized in Table 11.

TABLE XI. LINEAR COEFFICIENT OF THERMAL EXPANSION (α) FOR POLYIMIDE PWB MATERIALS

Board	Average (-55°C to +100°C) (Inches/Inch-°C)	Average (25°C to 150°C) (Inch/Inch-°C)
Epoxy	58.7×10^{-6}	131.0×10^{-6}
Experimental	21.6×10^{-6}	25.2×10^{-6}
A	47.7×10^{-6}	56.5×10^{-6}
B	47.1×10^{-6}	55.3×10^{-6}

The graphs of displacement versus temperature from which these data are calculated are very linear through -55°C to 100°C for the polyimide materials. However, this range encompasses the glass transition (Tg) of the epoxy, occurring at 90-100°C. Below this temperature, the α (epoxy) is comparable to those for the polyimides A and B.

b. Thermal Conductivity. The thermal conductivity of the polyimide boards was measured from 25°C to 150°C with a Dynatech Model TCFCM-N20 instrument using the comparative method. The values were obtained from 25-100°C, from which an average number for thermal conductivity was calculated.

TABLE XII. THERMAL CONDUCTIVITY VALUES FOR POLYIMIDE PWB MATERIALS

Board	T. C. (Cal/Sec/cm^2/°C/cm)
Epoxy	4×10^{-4}
Experimental	11×10^{-4}
A	3×10^{-4}
B	5×10^{-4}

c. Thermal/Vacuum Outgassing. The rate at which the polyimide products lost weight on heating in a hard vacuum was measured by a Cahn electro balance assembly contained in a vacuum bell jar. The pressure was reduced to at least 10^{-6} torr and the sample was heated on the balance at the rate of 2.0°C/minute from 25°C to 100°C. The outgassing rate was determined from weight loss data plotted on a recorder. The data are presented as a function of time and area in Table 13.

TABLE XIII. OUTGASSING RATES FOR POLYIMIDE PWB MATERIALS

Board	Rate (Weight Percent/CM^2/Hour)
Epoxy	0.14
Experimental	0.16
A	0.12
B	0.51

This test was performed in accordance with the MSFC Document 50M02442, "ATM Material Control for Contamination Due to Outgassing."

d. Flammability. The flammability of the polyimide PWB materials was determined in accordance with MSFC-SPEC-101B. This specification has provisions for testing combustible materials in oxygen-enriched environments as well as standard atmospheric oxygen contents. The specimens for this test were 2.5 inches (6.35 cm) by 12.0 inches (20.48 cm), and were ignited by a silicone igniter using bottom ignition so that the test involved an upward propagation rate. The three polyimide boards were tested in 100% gaseous oxygen at 6.2 psia and then in normal air using this method, and the results are given in Table 14.

TABLE XIV. FLAMMABILITY CHARACTERISTICS OF POLYIMIDE PWB MATERIALS

Board	100% O_2	Air
Experimental	Pass	Pass
A	Fail	Pass
B	Fail	Pass
Epoxy (Type GE)	Fail	Fail
Epoxy (Type GH)*	Fail	Pass

*GH: Heat resistant, fire retardant class of epoxy laminate

In this test the sample is considered to pass if, on upward propagation, the flame self-extinguishes within 6 inches (15.24 cm) from the bottom of the sample.

e. Water Absorption. The water absorption of the polyimide PWB samples was measured by submerging preweighed, 1 inch square (6.45 cm^2) samples in distilled water for 24 hours at 25°C. The samples were removed from the water, patted dry with a paper towel and reweighed on an analytical balance. The weight change in percent was used to represent the water absorption as shown in Table 15. This table also reflects the water absorption at elevated temperature.

TABLE XV. WATER ABSORPTION FOR POLYIMIDE PWB MATERIALS

Board	H_2O Abs. After 24 Hrs./25°C (Wgt. %)	H_2O Abs. After 1 Hr./100°C (Wgt. %)
Epoxy	0.496	0.176
Experimental	0.336	0.128
A	0.217	0.114
B	1.08	0.648

C. Thermal Cycling Studies

This section of the polyimide PWB evaluation deals with assessment of the effect of expansion normal to the plane of the boards on solder joint integrity during thermally-induced stressing of the joints. For this test sequence, three boards each representing the epoxy, experimental polyimide, and commercial polyimides A and B, were processed and etched in accordance with the previous section on processing. An appropriate test circuit pattern was used to allow a wide range of components and joint configurations. Each test board was 3.5 by 4.5 inches (8.89 by 11.43 cm) and contained the following components or joint terminations:

10 Resistors, RC07, 1/4 watt
16 Capacitors, kidney
2 Relays, 8-lead
1 Integrated circuit, flat pack
1 Integrated circuit, dual in-line
10 Transistors, TO-5
8 Transistors, TO-18
21 Jumpers, brass wire, 22 gage
21 Jumpers, copper wire, 22 gage
1 Connector, 23 pin
20 Terminals, 7-TAT-2
20 Terminals, 1-TAT-2

The components were soldered to the etched boards in accordance with standard MSFC soldering/techniques for flight hardware. Each board thus assembled contained a total of 337 soldered connections, or a total of 1,011 joints from each board type. The terminals were soldered on each side of the boards to provide the potential for a highly stressed joint. The two sides of a typical assembled test board are shown in

Figure 19. The boards were subjected to a pre-test microscopic inspection to assure the initial integrity of each solder connection. A thermal cycling test was then initiated, consisting of repetitive cycles of approximately 1.5 hours in duration providing temperature extremes of -55°C to +100°C. A typical cycle profile is as follows:

(1) 25°C to 100°C in 15 minutes

(2) Soak at 100°C for 10 minutes

(3) 100°C to -55°C in 30 minutes

(4) Soak at -55°C for 10 minutes

(5) -55°C to 25°C in 15 minutes

In-test microscopic examinations of each joint on each board were made at the end of 4, 10, 25, 51, 105, 152, 200, 340, and 500 cycles, after which the test was terminated. The effects of the cyclic temperature exposure on the joints were recorded as either cracking or other evidence of stress, according to joint configuration. The solder connections were formed from a variety of joint configurations on the board as listed below:

(1) Small terminals (on and off-pad terminations)

(2) Large terminals (on and off-pad terminations)

(3) Feed-through jumper wires (on and off-pad terminations)

(4) Connector terminals

(5) Dual in-line leads

(6) Transistor leads (on and off-pad terminations)

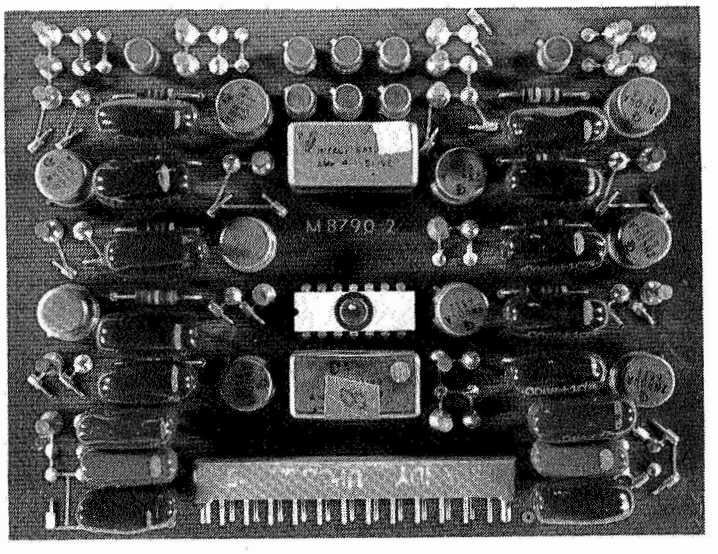
(7) Capacitor leads (on and off-pad terminations)

(8) Resistor leads (on and off-pad terminations)

(9) Relay terminals

(10) Flat pack Leads

Of these configurations, only items (1), (2), (3), (4), and (5) reflected significant joint cracking. A series of graphs, 20 through 25, reflect the incidence of cracking as a function of number of cycles for these five configurations. In each graph the number of cracks



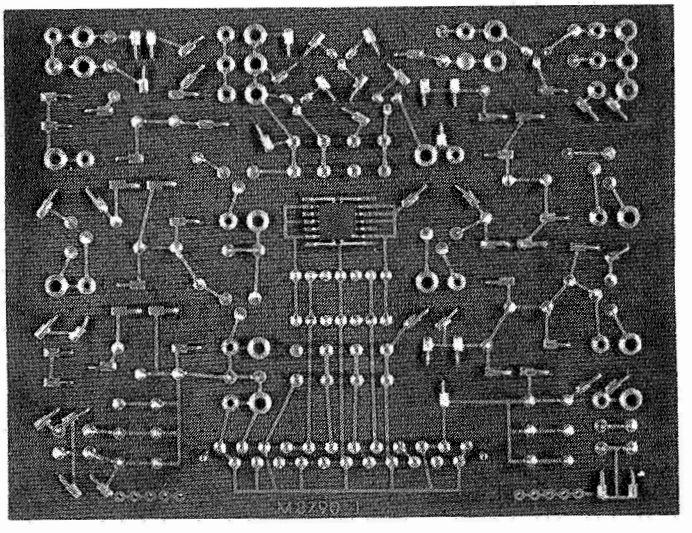


FIGURE 19. TYPICAL POLYIMIDE TEST PWB

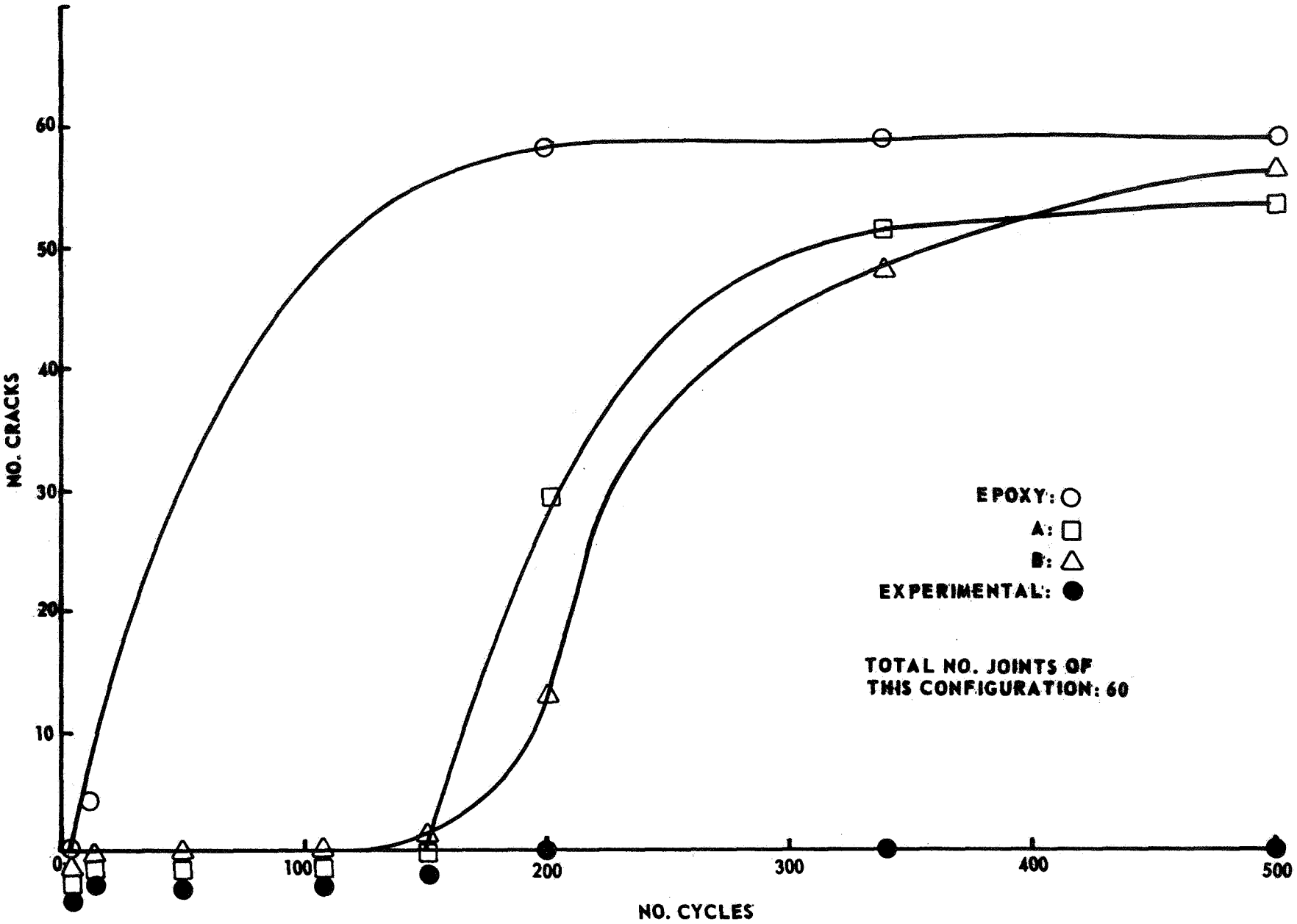


FIGURE 20 NO. CRACKS VS. NO. CYCLES FOR LARGE TERMINALS--SWAGE SIDE

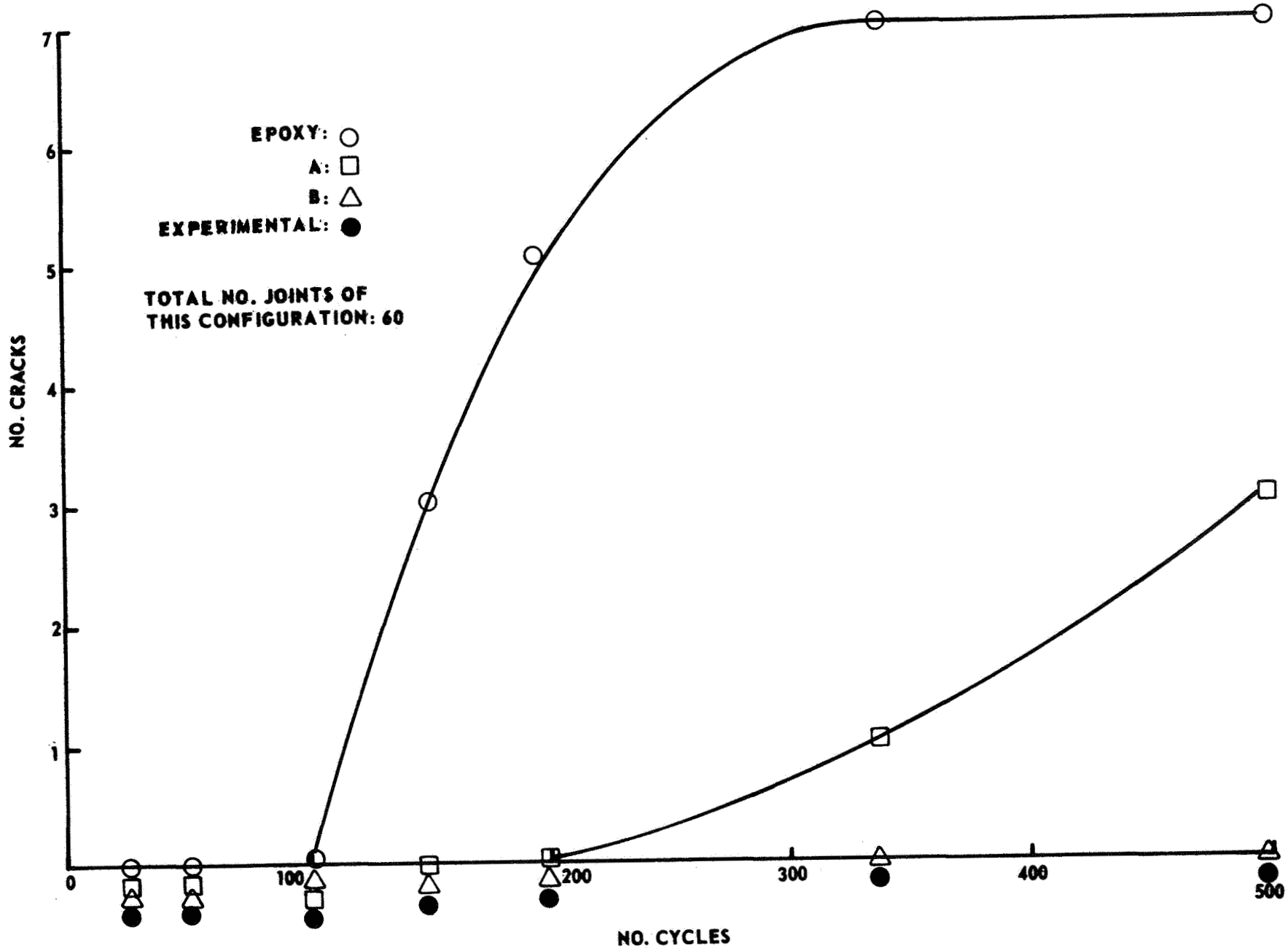


FIGURE 21 NO. CRACKS VS. NO CYCLES FOR LARGE TERMINALS–POST SIDE

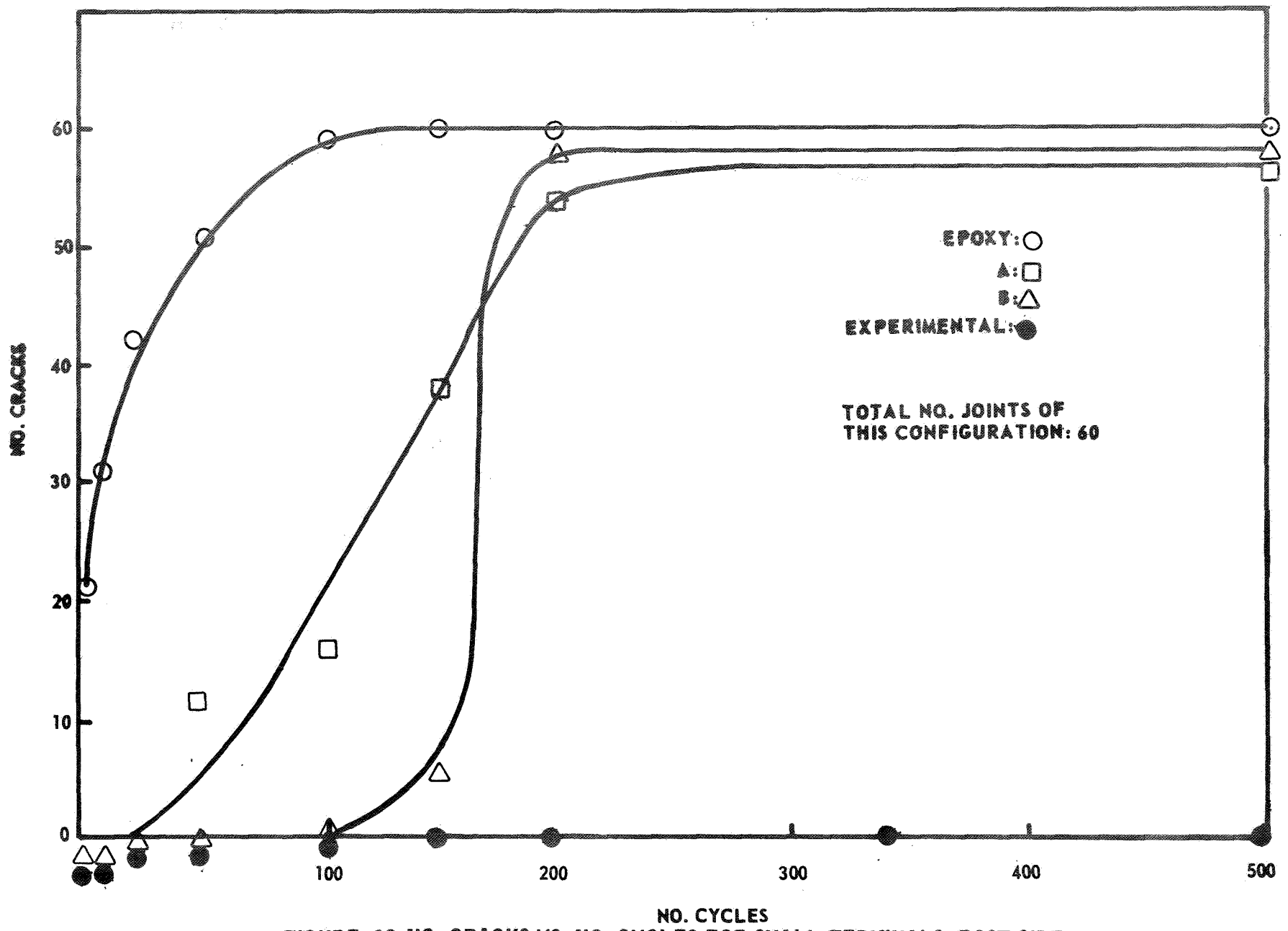


FIGURE 22 NO. CRACKS VS. NO. CYCLES FOR SMALL TERMINALS–POST SIDE

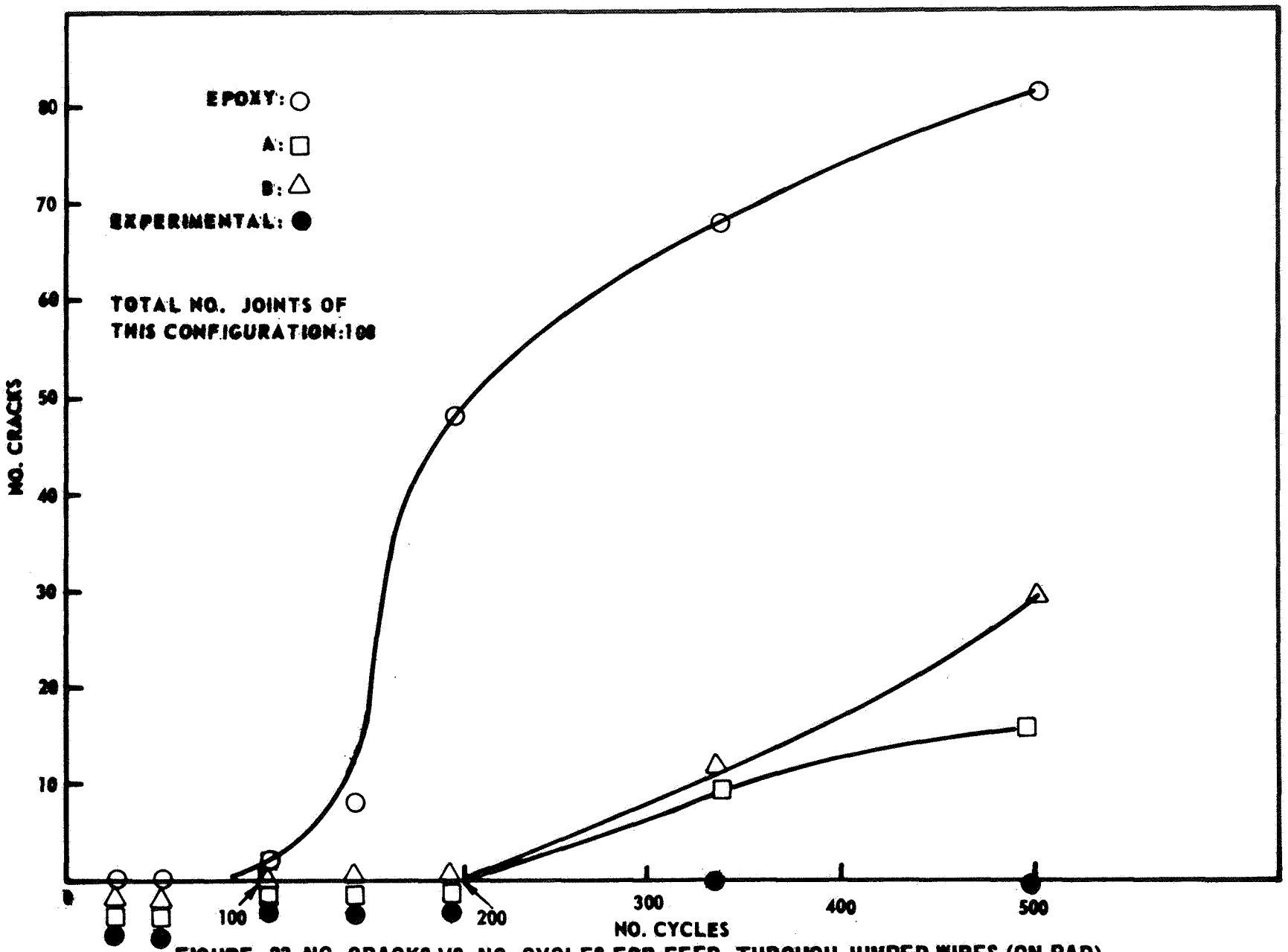


FIGURE 23 NO. CRACKS VS. NO. CYCLES FOR FEED-THROUGH JUMPER WIRES (ON PAD)

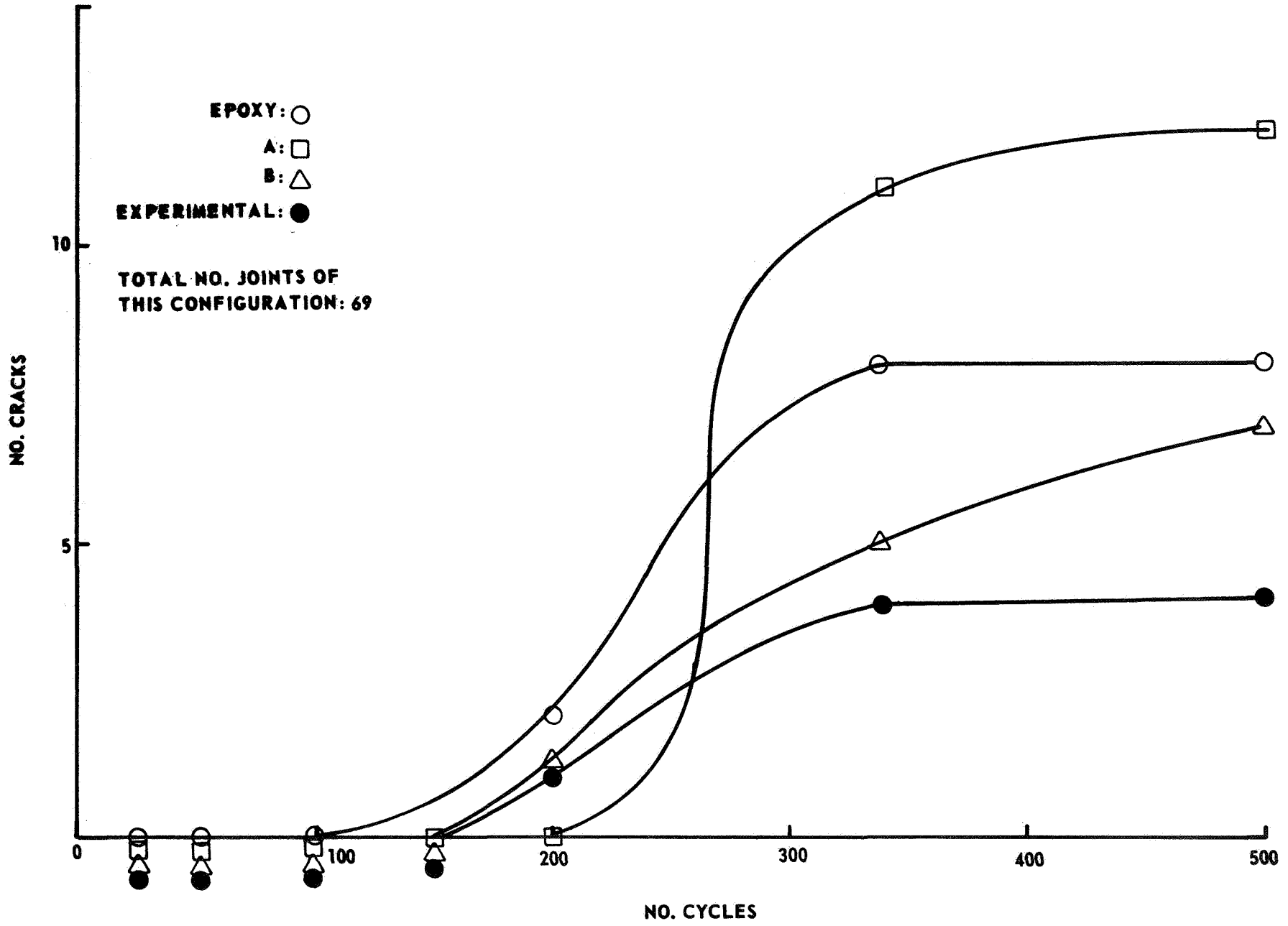


FIGURE 24 NO. CRACKS VS. NO. CYCLES–CONNECTOR TERMINALS

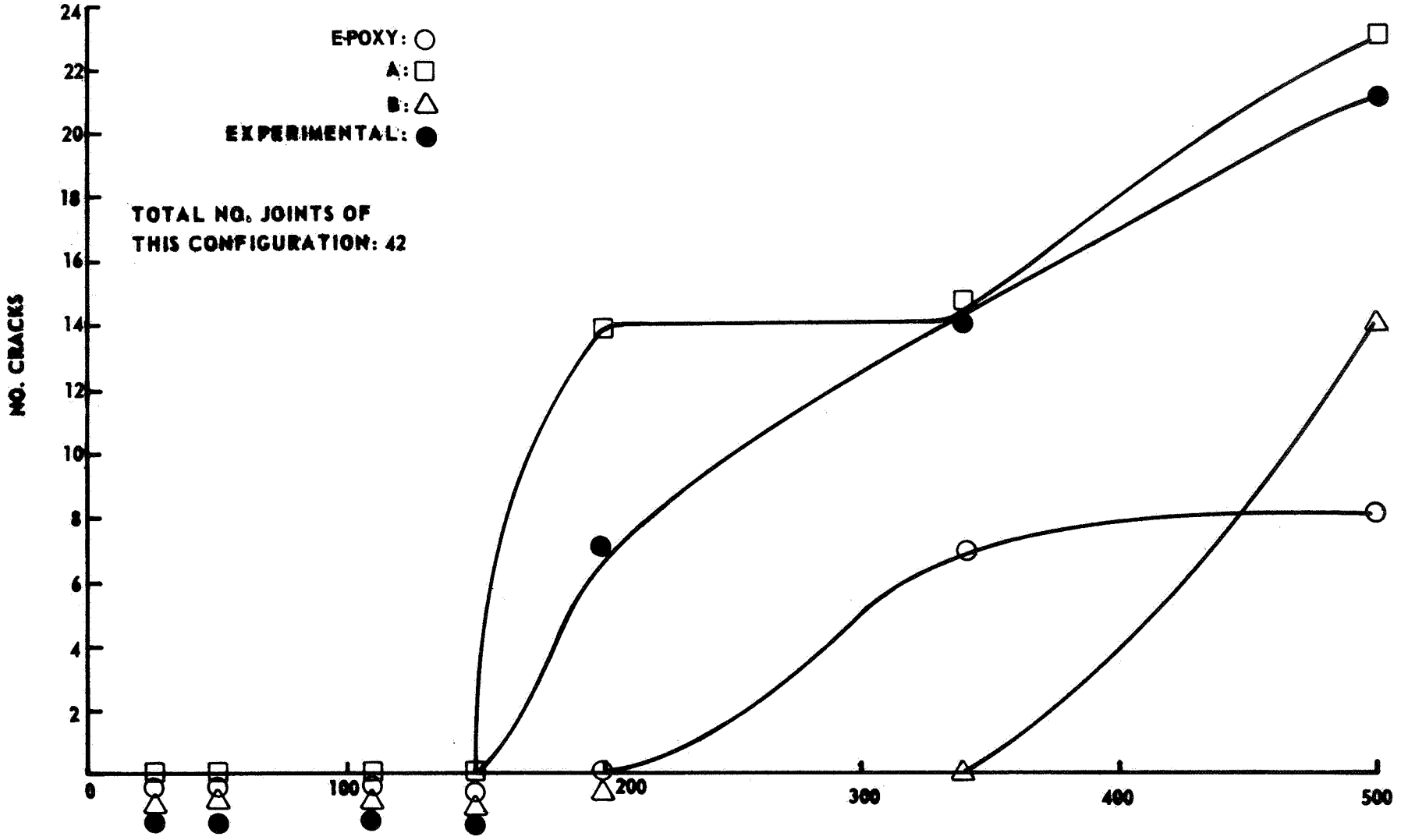


FIGURE 25 NO. CRACKS VS. NO. CYCLES FOR DUAL IN-LINE LEADS

plotted represents a sum over all three boards of a particular type. Also indicated on the graph is the total number (from three boards) of joints of the particular configuration being plotted.

The epoxy board samples were expected to reflect solder joint cracking early in the test, due to the high expansion coefficient of this material normal to the plane of the board, and this trend was observed in the test data as illustrated in Figure 20. The cracking initiated in these boards at some point between cycles 1 and 4, when the first inspection was made. The heaviest incidence of cracking for the epoxy and commercial boards A and B was observed in the terminal joint configurations illustrated by Figures 20, 21, and 22. The outstanding exception to this trend was the experimental board which had no cracked joints for any terminal configurations through the 500 cycle test duration. Of the two commercial polyimides, board B appears to contribute less to joint cracking for this configuration. The same general trend is observed for the 4 board types for the feed-through jumper wires (Figure 23). In the case of connector terminals and dual in-line leads the trend changes and the epoxy is no longer the worst offender (Figures 24 and 25).

The total number of cracks from every joint configuration as a function of cycling is depicted in Figure 26. Again the number of cracks represent a sum over 3 boards for each board type. This information is presented in percentage form in Table 16, based on the incidence of cracking in each board material after 500 cycles.

TABLE XVI. PERCENT CRACKS FROM ALL CONFIGURATIONS AFTER 500 CYCLES FOR POLYIMIDE PWB MATERIALS

Board	% Cracks*
Epoxy	21.8
Experimental	2.47
A	15.8
B	16.3

*Based on total values summed over 3 test boards of each type

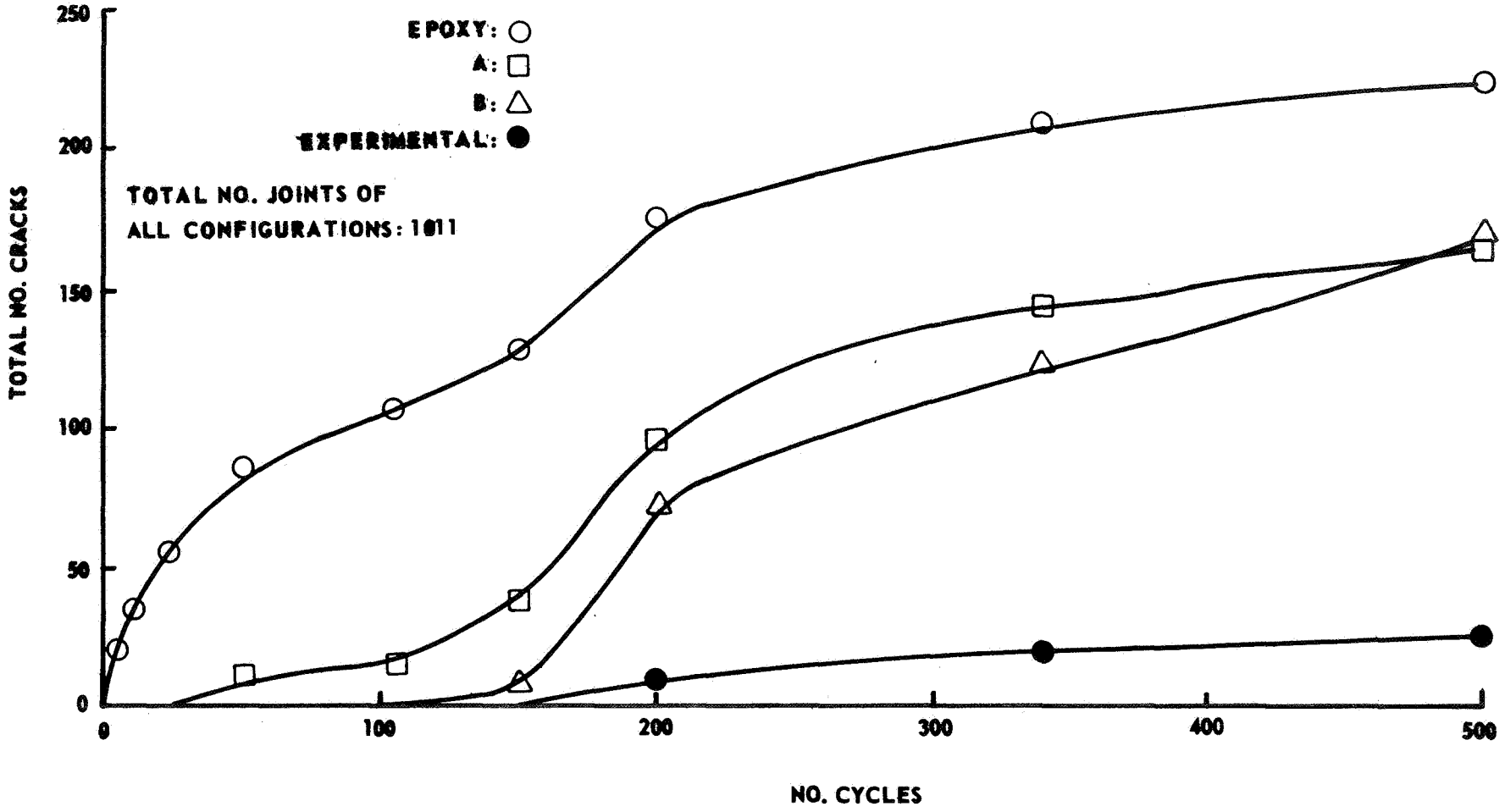


FIGURE 26 NO. CYCLES VS. TOTAL NO. CRACKS FOR ALL JOINT CONFIGURATIONS

The photographs in Figures 27, 28 illustrate the severity of cracking in various joint configurations. The condition in Figure 29 could quite easily represent an electrically isolating crack that would lead to circuit failure.

DISCUSSION

A. Experimental Board Fabrication

The basic design of the experimental polyimide board has been shown to result in the desired program goals, especially in the critical area of expansion normal to the plane of the board. It is believed that the particulate filler and the polyimide (with no polymer glass transition below 100°C) provide nearly the entire control of expansion of the PWB in this axis. The tridirectionally woven cloth, when studied from photomicrographs of sectioned laminate, has little orientation normal to the laminate plane and is considered to exert proportionately less control over expansion.

The basic materials selection for the experimental board is expected to allow standard PWB processing. All these materials are commercially available as standard items without special ordering or fabrication. The polyimide resin is processed by conventional prepreg techniques. The main departure from standard laminate technology is in introduction of the particulate filler. At 20 percent by volume filler loading, a non-settling dispersion forms which results in homogeneous deposition on the cloth at the prepreg stage. The silane additive in this mixture promotes good adhesion of the resin matrix to the filler. The 316°C laminating temperature for the laminate-forming process is quite high relative to normal epoxy technology, but has become accepted by polyimide laminate producers as a working condition that can be met using high temperature presses. In general, the experimental board is seen as a product that is commercially feasible with little departure from normal polyimide processing technology.

B. PWB Evaluation Analysis

Acceptable drilling techniques have been devised for polyimide boards. Two precautions which must be followed are the reduction in drill speed to the range of 600-2500 rpm and use of a hard back-up material such as G-10 epoxy laminate for the drilling operation. Better copper foil/laminate bond integrity is also obtained by using a processing sequence of drilling before etching. In this manner, the entire foil layer is available to resist debonding during drilling, rather than just the solder pad. While test coupons such as the one in

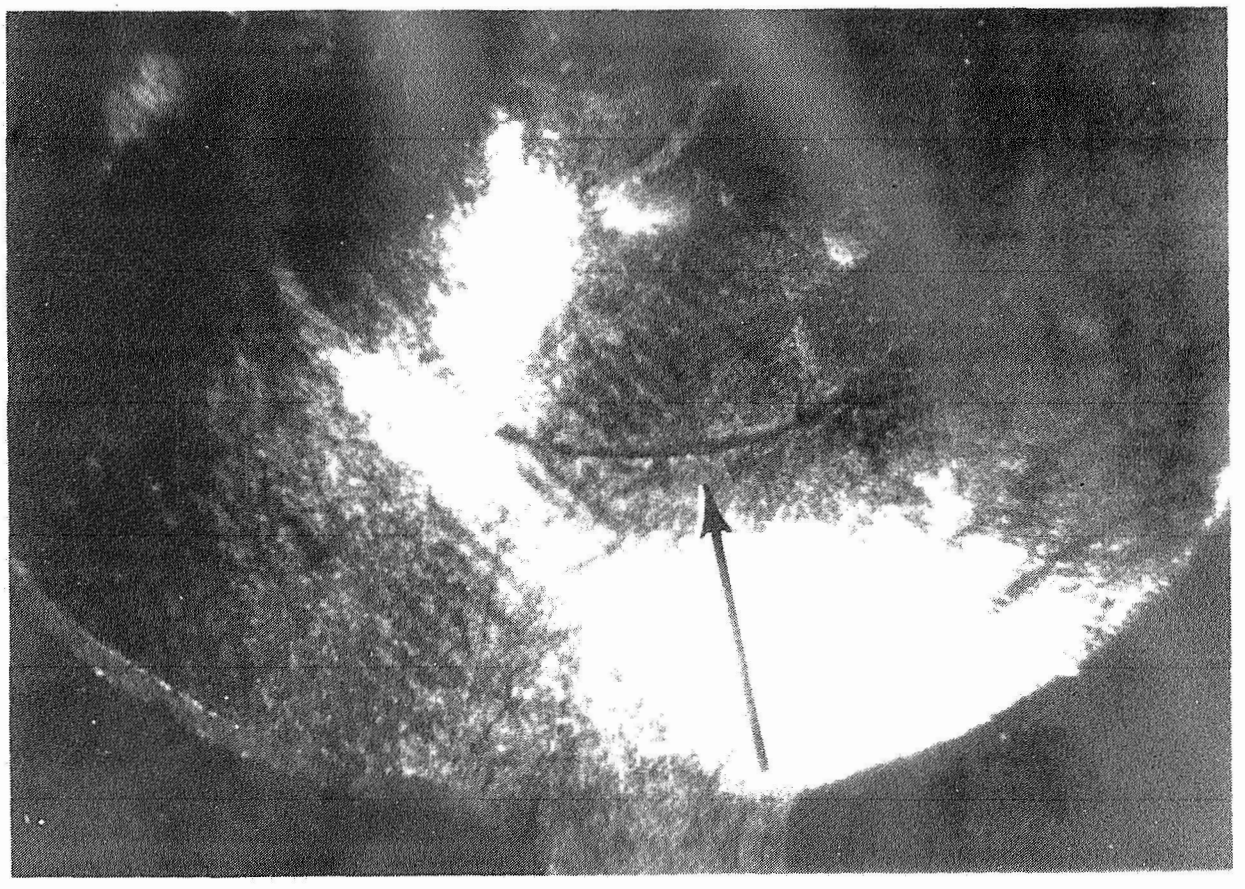


FIGURE 27. CRACKED SOLDER CONNECTION AFTER 4 CYCLES FROM EPOXY PWB, SMALL TERMINAL (20X)

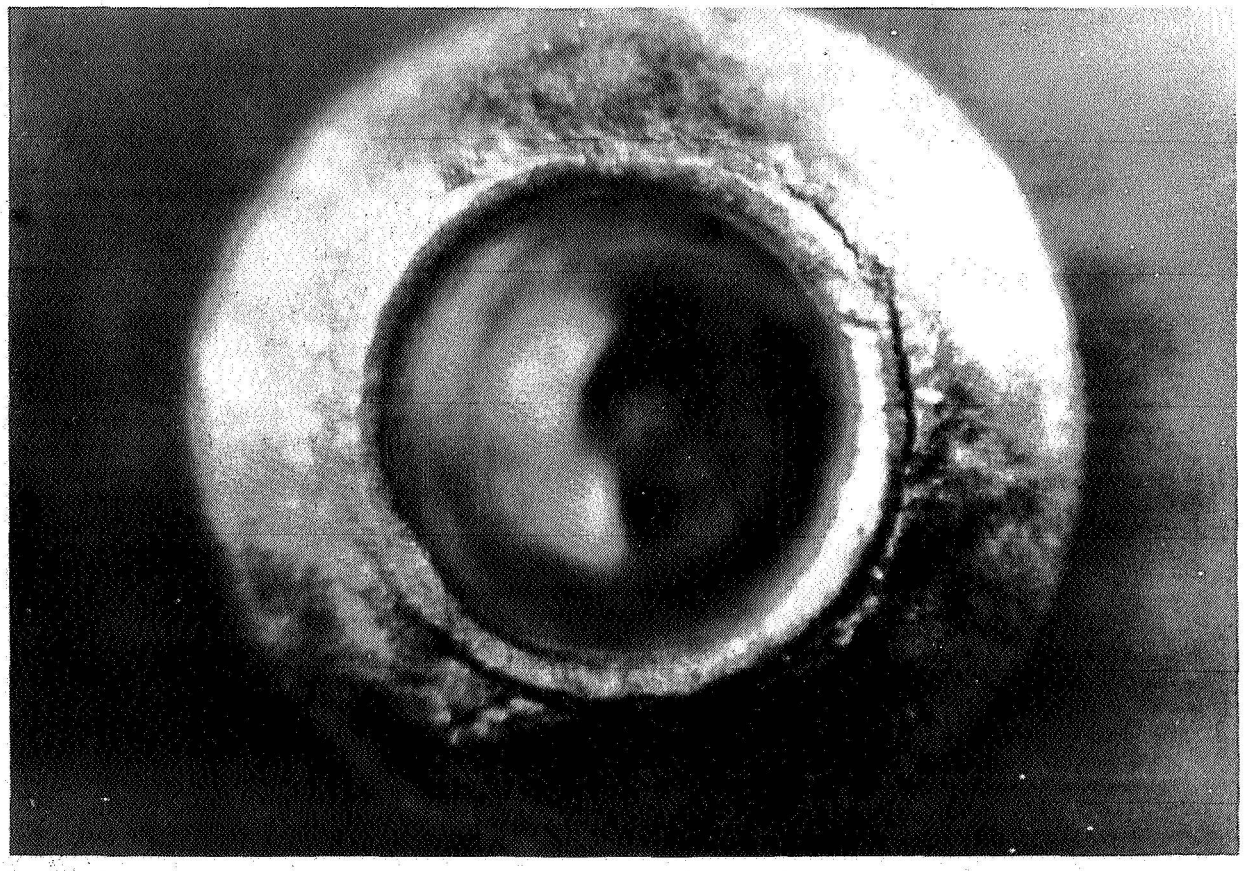


FIGURE 28. CRACKED SOLDER CONNECTION AFTER 500 CYCLES FROM EPOXY PWB, LARGE TERMINAL (15X)

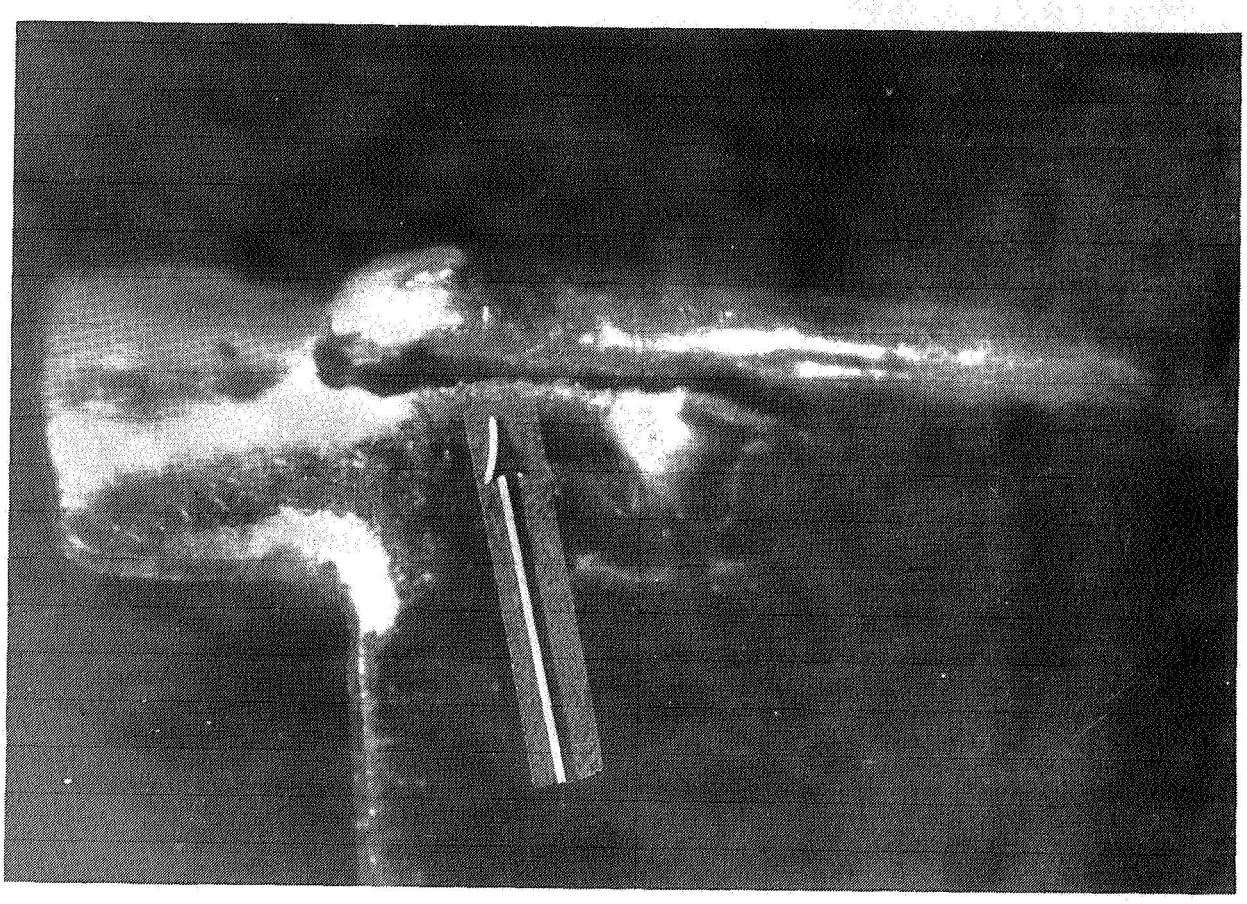


FIGURE 29. CRACKED SOLDER CONNECTION AFTER 105 CYCLES FROM COMMERCIAL PWB B (15X)

Figure 7 indicate adequate bond of the solder pad to withstand the drilling operation, the polyimide laminate is normally more susceptible to resin-rich and resin-poor areas than is the epoxy laminate, so that the copper/laminate bond strength might vary considerably. Thus the drilling sequence and back-up sheet precautions are advisable as standard processing operations.

All of the polyimide boards can be processed by standard epoxy PWB technology with one important deviation. It is essential that exposure to hot, alkaline solutions be limited to very short periods. Continued exposure would hydrolyze the imide ring in the polymer backbone back to its precursor, the polyamic acid, which could undergo further base-catalyzed hydrolysis to give diacid and amine fragments as products of chain cleavage. This last process represents catastrophic degradation of the polymer by significantly lowering its molecular weight and, concomitantly, its mechanical and physical properties. Cleaning or plating processes involving polyimides should severely restrict the use of alkaline materials.

In comparative evaluation of mechanical properties of the polyimide materials relative to the type G-10 epoxy PWB, an outstanding advantage of the polyimide material in general is its relative independence of mechanical properties on temperature over the operating range of most electronic hardware. As evidenced in the copper peel strength values in Table 4, the epoxy PWB peel strength has a higher room-temperature value than does the polyimide, while at 150°C the polyimide strength is 77.9-99.1 N/M (8.8-11.2 pounds/inch) but the epoxy peel strength dropped to 36.3 N/M (4.1 pounds/inch), which represents marginal adhesion. This has important implications in PWB rework, since the desoldering operation to replace components necessitates heating the joint and the solder pad to 180-185°C to melt the solder, and provides the mechanism for debonding the solder pad from the board. Thus retention of copper/board adhesion at solder melting temperatures is quite important. The values at 260°C in Table 4 are given as greater than 53.1 N/M (6.0 pounds/inch) because tensile failure of the copper strip occurred at this temperature rather than adhesive failure to the laminate. The peel strengths of the polyimides are projected to be slightly greater than 53.1 N/M at 260°C. The experimental board held up adequately through the various environmental exposures although not as well as boards A and B. The terminal pull test reflected the same trend as shown in Figure 10, where the epoxy initially required a higher tensile force to pull off the soldered lead, but this value dropped significantly between 75 and 100°C coinciding with the glass transition range of the epoxy laminate. This is not a fortuitous event, since transitions of this type in polymers are sometimes accompanied by gross changes in mechanical properties, among them being adhesion. In the terminal pull test, the limiting factor was not loss of adhesion but rather melting of the solder (eutectic solder, 63/37 SN/Pb, melts

at 183°C). The flexural strength tests again reflected this trend, with the epoxy laminate decreasing substantially in flexural strength in the region of its glass transition.

The electrical test results generally indicate that the polyimide boards are equivalent to the type G-10 epoxy material. One important exception to this trend is the improved stability of dielectric constant and dissipation factor of the polyimide as a function of temperature or frequency. At the risk of boring the reader, the glass transition of the epoxy is again indicted as a basis for this material's temperature-dependent dielectric constant and dissipation factor as illustrated in Figures 15 and 16. Above the glass transition temperature of the epoxy, the molecular motion of side chains and that of the main polymer backbone increases tremendously, resulting in increased dipole interaction and dielectric loss. By comparision, the dielectric properties of the polyimides are affected only very slightly over the range of 25-175°C. The numerical value of dielectric constant for the experimental polyimide is somewhat higher than A and B, although its dissipation factor is lower.

The insulation resistance of the three polyimides is quite comparable, both at ambient conditions and after environmental conditioning. A slight improvement is observed over the epoxy PWB.

Volume and surface resistivity, dielectric withstanding voltage, arc resistance and dielectric breakdown measurements on the four test materials show comparable numerical values, with no single material consistently better than another.

Perhaps the most revealing property measurement in the entire PWB evaluation section is that of linear coefficient of thermal expansion (α). The measurements were made quite accurately in an axis normal to the plane of the boards using the automated thermal analysis equipment described in a previous section. The primary α-values were calculated as an average over the range of -55 to +100°C to coincide with the thermal cycling studies. As indicated in Table 11, the experimental board has an α-value over twice as low as either commercial polyimide, and this trend holds for both temperature ranges. The disproportionate increase in the α (epoxy) over the higher range reflects the effect of the glass transition in that material. The polyimides are all characterized by a reasonably linear increase in expansion with temperature up to approximately 200°C, where all three materials begin a greater expansion rate with temperature. The lower expansion coefficient for the experimental polyimide is reflected quite vividly in the thermal cycling test results.

The results of the thermal conductivity measurements also point to an improvement in the case of the experimental board. The approximately

two-fold increase in thermal conductivity is apparently due to a much more efficient thermal path created by the alumina particles. This property allows the PWB to function more effectively as a thermal sink to dissipate heat from heat-generating components.

The thermal/vacuum weight loss tests were carried out on the polyimides to determine if they contained a significant amount of volatile materials which might recondense on critical optical surfaces of experiments or equipment in space. The epoxy as well as the experimental board and board A were within the acceptable outgassing levels established in MSFC Document 50M02442 for critical optical surfaces. The polyimide B was considerably higher and this is consistent with the moisture absorption data which also reflected a higher moisture absorption for polyimide B. The weight loss in materials of this type can generally be ascribed either to removal of volatiles produced in the laminate-forming step or from atmospheric moisture permeating the somewhat more porous structure. This porosity would also explain the increased moisture absorption in B.

The flammability tests performed on the polyimides were those used to qualify materials for use in 100% gaseous oxygen environments aboard the Skylab workshop. The experimental board alone passed this test, which implies a lower organic resin content. This is obviously the case, since the inclusion of 20% filler reduces the overall polyimide resin content relative to the other boards. The commercial boards A and B readily passed the test when conducted in air.

The results of the thermal cycling studies carried out on the test boards serve generally to reinforce the conviction at the beginning of this program that effective control of expansion in the axis normal to the plane of the board would significantly reduce PWB solder joint cracking. The experimentally measured values of α in Table 11 represent very nearly a direct correlation with the cycling test results presented in Table 16 and in Figure 26.

There are two seemingly anomolous test results, in the connector terminal and dual in-line joint configurations. Referring to Figure 24, all boards show cracking after 200 cycles for the connector configuration, although the experimental still has the lowest overall incidence of cracking. The connector body is normally constructed of molded nylon or diallyl phthalate, both of which are relatively high-expansion materials. The connector body is pulled flush against the PWB surface and constrained by the solder connection. Thus a certain amount of stress should be transmitted to the solder joint through tensile and compressive loading of the lead by the high-expansion connector body during thermal cycling. This obviously is not a function of the expansion characteristics of the PWB, and the connector configuration data should be assessed with this in mind.

In the case of the dual in-line configuration data (Figure 25) the experimental board had a relatively high incidence of cracking. This condition seems to have no satisfying explanation to date. The leads were not soldered on the component side, and the component was well up off the board so the board could expand and contract around the leads without transmitting stress to the joints. This anomoly will be pursued in further studies.

CONCLUSIONS

This development program has culminated in a new, low-expansion polyimide PWB which minimizes solder joint cracking due to board expansion and contraction. The experimental board is fabricated from commercial materials with state-of-the-art polyimide laminate technology. With the exception of restrictions on drilling and alkaline solution exposure, the polyimides adapt reasonably well to epoxy PWB technology. In terms of mechanical, electrical, and thermal properties, the polyimides are equal or superior to the epoxy in nearly every instance.

Now, in comparing the three polyimides the message from the thermal cycling study is that the commercial boards A and B do not have a sufficiently low α normal to the plane of the board to result the needed improvement over G-10 epoxy performance. Of the materials investigated in this study, only the experimental board provides the very low incidence of solder joint cracking required in high reliability space hardware electronics.

APPENDIX

PWB PRODUCTION

I. Materials and Sources

A. P13N Polyimide Resin
TRW Systems, Redondo Beach, California
Geigy Chemical, Ardsley, New York

B. GT treated Copper Foil
Circuit Foil Corporation, Bordentown, New Jersey

C. 7628 Fabric
Burlington Glass Fabrics, New York, New York

D. Tricon HS-1 Fabric
A. Wimpfheimer & Brothers, Inc., Stonington, Connecticut

E. Alumina (A-14) Hard Fired, Ball Milled
Alcoa

F. A-1100 Coupling Agent
Union Carbide, Silicone Division
New York, New York

II. Impregnating Systems

A. Resin: TRW P13N Polyimide, 40 ±2.0 percent solids
Solvent: Dimethylformamide
Viscosity at 25°C: 200-300 cps

B. Copper Primer:
P13N Polyimide resin - 2 mil dried coating

C. Fabric Impregnating System
Resin: 40 percent solid ----------75.20 weight percent
Alumina (A-14) ---------- 24.03 weight percent
A-1100 ---------- 0.77 weight percent

Any mixing procedure with sufficient shear to break up powder agglomerates can be used. Experimental systems have been homogenized by rolling a can containing ceramic balls on slow-speed rolls. Larger quantities have been blended using a dispersator high-speed mixer, however, care must be taken to avoid heating the system.

III. Coating and Impregnating Procedures

A Dixon coater was used for coating both the 7628 and HS-1 fabric. The coating traverse and conditions used are shown in Figure 30. Good wetting and pickup was obtained with this system. Nip-roll settings of 15 and 31 mils were used for the 7628 and HS-1 reinforcements respectively. Maximum dwell time (15 minutes) was adequate to dry the system to touch, but not to a tack-free state. Consequently, a thin Mylar parting film was fed into the roll in the rewind step. Some tacking to the Teflon-coated rolls was experienced due to the slow drying characteristics of the solvent. Accordingly, it is believed that a 30 foot or higher vertical oven would perform somewhat better with this system. Although recommended drying time for the P13N resin is 15 minutes 60°C, experience has shown that blistering occurs unless a longer drying time is used. In the absence of a continuous unit for completion of the drying and prepreging step, these reinforcements were sectioned into sheets 12-1/2 x 12-1/2 inches for subsequent laminating. A number of sheets were suspended on a light-weight metal frame and placed first in a 60°C oven for 30 minutes followed by 1 hour in a 204°C oven.

Laminating sized sheets of copper (12-3/4 x 12-3/4 inches) were slipped to light-weight metal plates, coated, and placed first in a 60°C oven for 1/2 hour followed by 1 hour in a 204°C oven. No difficulty was noted in the processing or handling of either the impregnated fabrics or the coated foil which would preclude the use of continuous units with the necessary range of speeds and temperatures.

IV. Laminating Procedures

A. Sheet preparation--None was needed. However, these may be preflattened, if necessary, by lightly pressing in a 150°C press. (Conventional impregnation and drying procedures provide adequate constraint that this precaution is unnecessary.) Sheets are trimmed to a square pattern.

B. Lay-up--This consists of a three-ply HS-1 core and a single ply of 7628 cloth on each side. Each ply is rotated 90° from the previous ply in the stack. The lay-up is topped with the precoated copper foil.

C. Stabilization--Small sized laboratory lay-ups including the 12-1/2 x 12-1/2 inch laminates were tied to prevent shifting. Larger sized lay-ups should have adequate contact area to prevent significant shift during pressing.

D. Isolation plates--1/32 inch polished stainless steel sheets.

E. Preheat and volatile reduction--Contact pressure in a preheated (343°C) press for time necessary to degas and heat matrix. Time is highly dependent upon heating rate, and rapid heating appears desirable. Press controls reduced to 316°C before insertion of laminate. (Contact time may be as short as 30 seconds or as long as 6-1/2 minutes depending upon heating rate.)

F. Lamination--Raise pressure to 1000 to 1500 psi and hold 1 hour at 316°C.

G. Board may be cooled under pressure or pulled hot. It is advisable, however, to keep the stack intact to minimize thermal shock and possible warping.

V. Specifications

A. Matrix: Development studies indicate a good balance of thermal properties are achieved at a filler level of 22.5 volume-percent.

B. Prepreg: Laminate fabrication and properties appear to reach an optimum around 35 w/o matrix in the fabrics. Accordingly, impregnation is targeted to this value.

C. Copper primer: Two mils of P13N after drying and prepreging is desired.

D. Laminate: When fabricated with the following components:,

Copper:	Two faces - 2 mils neat resin
7628:	Two plies - 35 w/o matrix
HS-1:	Three plies - 35 w/o matrix

and laminated as described earlier, the thickness falls within the tolerances of the G-10 specifications.

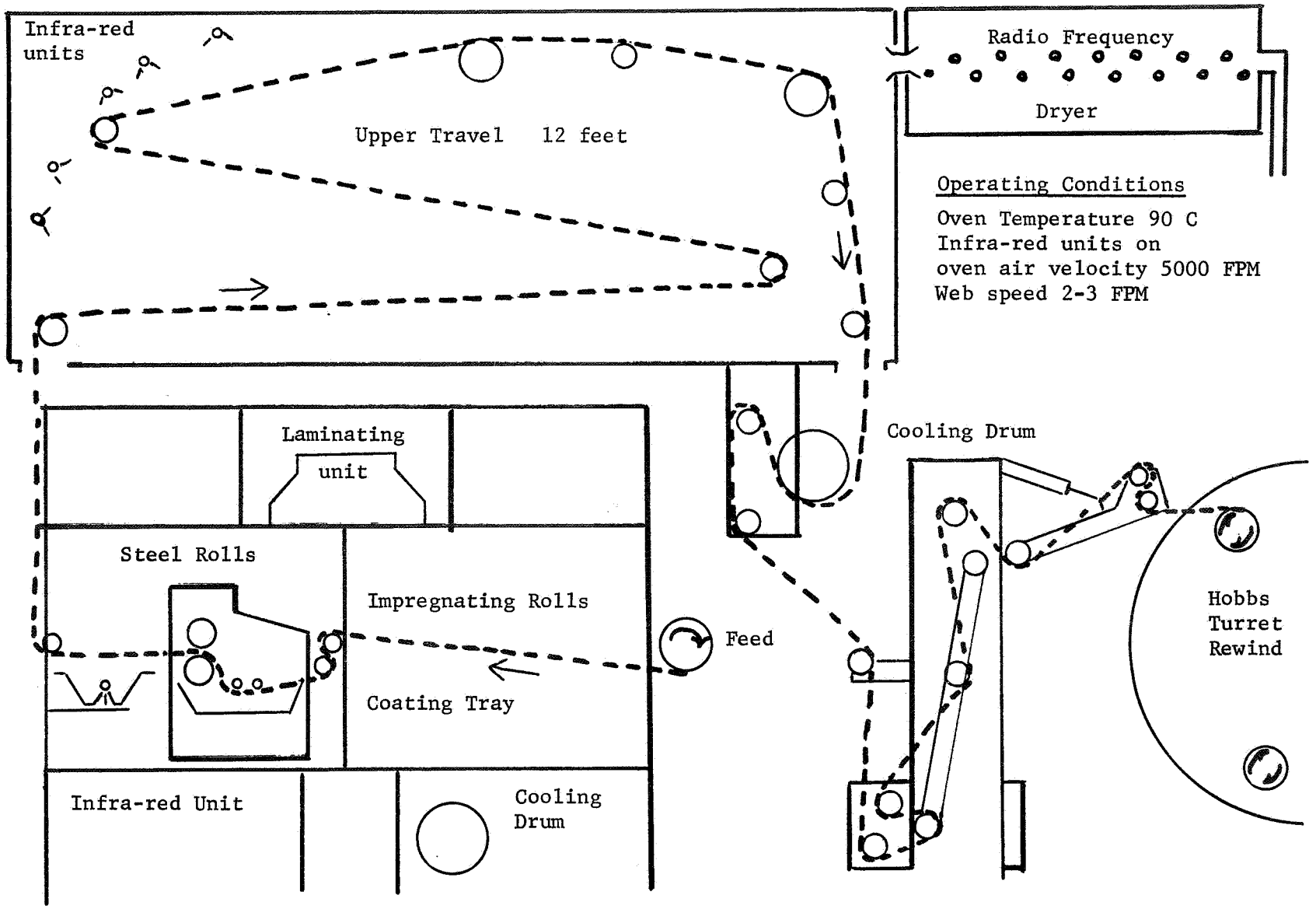


FIGURE 30. PREPREG FABRICATION FOR EXPERIMENTAL POLYIMIDE PWB

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APPROVAL

DEVELOPMENT AND EVALUATION OF
POLYIMIDE LAMINATES FOR PRINTED
WIRING BOARD APPLICATIONS

By

William J. Patterson

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commision programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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